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RESEARCH MEMORANDUM

EFFECT OF SURFACE-ACTIVE ADDITIVES ON PHYSICAL
BEHAVIOR OF 50-PERCENT SLURRIES OF 1.5-MICRON
MAGNESIUM IN n-DECANE

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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

WASHINGTON

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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

RESEARCH MEMORANDUM

EFFECT OF SURFACE-ACTIVE ADDITIVES ON PHYSICAL BEHAVIOR OF

50-PERCENT SLURRIES OF 1.5-MICRON MAGNESIUM IN n-DECANE

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SUMMARY

In a study of the use of surface-active additives to reduce the viscosity of thick slurries consisting of 50 percent by weight of 1.5-micron magnesium powder in n-decane, it was found that as the slurry became increasingly fluid the magnesium tended to settle more and to form a harder cake. In the most fluid slurries, the magnesium settled in a very hard cake, which made remixing difficult.

The viscosity of the slurry was dependent on the concentration and composition of the additive. When the molecules of additive were bulky and contained multiple polar groups, the viscosity decreased rapidly and continuously with increasing additive concentration. When the molecules of additive were linear and had only one polar group, the viscosity-concentration curve had a minimum. As the concentration of either type of additive was increased, the slurry changed gradually from a pseudo-plastic, thixotropic material to one which was plastic and nonthixotropic. In order to facilitate the work, most of the additives were evaluated at only one concentration, 1.00 percent based on the weight of slurry (except for three carboxylic acids).

Of the 50 additives investigated, the ones which yielded the most fluid slurries were those that had a hydroxyl group together with an ester, metal salt, or polyoxyethylene group in the molecule. Those which had only one of these polar groups gave slurries that were less fluid.

Other factors influencing the viscosity of the slurry were age, temperature, moisture, and the characteristics of the magnesium powder. Aging usually caused the viscosity to decrease and then to level off. The viscosity of the slurries increased as the temperature was decreased from about 140° to 0° F, the magnitude of the increase depending on the nature and concentration of the additive. Removal of part of the moisture (or perhaps other components of the air) from the magnesium powder prior to the addition of the n-decane also caused a decrease in the viscosity. However, essentially the same effect could be achieved more readily by the incorporation of a suitable surface-active additive.

A limited amount of data indicated that as the concentration of additive was increased, the plastic viscosity and apparent yield value either decreased continuously or reached a minimum, depending on the nature of the additive. Viscosity measurements with a Stormer viscometer and a Severs rheometer were of the same order of magnitude and much smaller than the Brookfield apparent viscosity.

A mechanism was proposed to explain the effect of surface-active additives on slurries, and suggestions were made as to the application of the results obtained in this investigation.

INTRODUCTION

An analytical evaluation (ref. 1) of metal-hydrocarbon slurries at the NACA Lewis laboratory indicated that magnesium-slurry fuels offer more thrust than do conventional jet-engine hydrocarbon fuels. Combustion tests carried out in experimental burners have confirmed this analysis and also have shown that magnesium-slurry fuels can be burned over a wider range of equivalence ratios than the conventional hydrocarbon fuels (refs. 2 to 4). The data reported in references 3 and 5 indicate that the combustion performance experimentally obtainable with these fuels increases progressively as the particle size of the suspended magnesium becomes smaller.

In previous reports (refs. 6 to 9) describing the physical properties of slurry fuels prepared by mixing magnesium powder with hydrocarbons, the magnesium consisted almost entirely of spherical particles of 15 to 20 microns average diameter. The principal objective was to increase the consistency of the very fluid slurry so as to retard the settling of the magnesium without adversely affecting the other characteristics. The use of gelling agents (aluminum soaps) for this purpose is discussed in references 6 and 7, and the use of petrolatum in reference 8. The effect of particle size on some of the physical properties of petrolatum-stabilized slurries is discussed in reference 9.

In contrast, slurries consisting of 50 percent very fluid hydrocarbon and 50 percent magnesium particles which average 2 microns or less in equivalent spherical diameter are pasty and do not flow readily. Such slurries are so difficult to pump or spray into a combustor that it is necessary to reduce their viscosity by incorporating a surface-active additive.

Since the use of surface-active additives to disperse solid particles in suspension is of great practical interest, especially in the paint industry, there have been published many evaluations of the relative effectiveness of various additives in systems different from the one under consideration in this report. Because the attractive force between the

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suspended particles and the molecules of additive is so important, and steric factors are probably also important, not all additives which might be surface-active are equally effective, and their effectiveness varies with the composition of the suspension. Thus, one investigator found that of 35 compounds added to titanium dioxide - n-heptane mixtures, calcium, lead, and zinc naphthenates, calcium petrosulfonate, and lead octoate were the most effective, while aniline and cyclohexanol had no effect (ref. 10). Another investigator has stated that it is difficult to predict what sort of surface-active agent will be the most useful in a paint (ref. 11), and this statement may presumably be applied to the magnesium slurries.

In the present investigation, which was begun in June, 1953, the objectives were to determine what types of compounds are surface-active additives for magnesium slurries and how surface-active additives affect some of the physical characteristics of slurries. The selection of additives that would be the most suitable and the concentration in which they should be used were also considered.

Slurries consisting of 50 percent by weight of 1.5 micron (average equivalent spherical diameter) magnesium powder and 50 percent purified n-decane plus additive were prepared and tested. This magnesium powder was of the smallest particle size available in adequate quantity at the time. Purified n-decane was used instead of one of the fuels of the JP-series (which would ordinarily serve as the hydrocarbon carrier) in order to avoid accidental introduction of surface-active compounds. The 50 additives evaluated were commercially available materials chosen so as to represent a wide variety of molecular structures. The Brookfield apparent viscosity, extent of settling, and degree of packing of the sediment of the slurries were measured. In addition, viscosity measurements were made on two typical series of slurries with a Severs rheometer and a Stormer viscometer. The effects of moisture and temperature on the apparent viscosity of the slurries were also investigated.

A glossary of certain terms and a summary of the pertinent literature are provided in appendixes A and B.

MATERIALS AND PROCEDURE

Materials

Magnesium powder. - The magnesium powder was prepared commercially by the atomization of 99+ percent pure magnesium in a helium atmosphere. Three drums of the powder were used; the contents of these drums will be referred to as magnesium A, B, and C. Data on the particle-size distribution and assay of the powder are given in table I and an electron micrograph of the powder is shown in figure 1.

[REDACTED]

Normal decane. - The n-decane was distilled in a packed 30-foot stainless steel column. The initial and final fractions were rejected, and the remainder was percolated through 4-foot glass columns packed with 20 to 65 mesh activated silica gel to remove polar impurities.

The physical properties of this n-decane and the corresponding values cited in the literature are listed in table II. The high purity of the n-decane is shown not only by the freezing and boiling points, but by the interfacial tension against water, which indicates the substantial absence of polar impurities.

Additives. - The 50 additives evaluated for surface activity are commercially available products and were used without modification. They will frequently be referred to by the code numbers listed in table III. Their approximate composition and some of their physical properties, as reported by the manufacturers or estimated from other information, are also listed in table III. The types of polar group in each additive are given for those additives whose structure is known.

With the exception of several proprietary materials of unknown composition, the additives were chosen on the basis of their molecular structure so as to represent a variety of different structures. When more than one slurry was prepared with a given additive, the same batch of additive was used throughout. When the additive was supplied in diluted form (see footnote b, table III), a sufficient quantity was incorporated into the slurry to give the desired concentration of active ingredient. Except for a small number of slurries for which data are presented in appendix C, only one additive was used in each slurry.

Preparation of slurries. - All the slurries were made in 400-gram batches and consisted of 50.0 percent magnesium powder and 50.0 percent n-decane plus additive by weight. With the exception of one series of samples, the slurries were prepared in new, 1-pint, tin-plated paint cans which had been successively rinsed with pure acetone and pure n-heptane and wiped dry with a clean cloth after each rinsing. The additive was weighed into the can to the nearest 0.01 gram, and then the n-decane was weighed into the can to the nearest 0.1 gram and stirred (and warmed, if necessary) to dissolve or disperse the additive. Finally, the magnesium powder was weighed into the liquid, and the mixture was vigorously stirred by means of a motor-driven stirrer while being heated on a hot plate to 140° to 160° F. After any evaporation loss was made up with more n-decane, the can was sealed and cooled in an 86° F bath. This procedure will hereinafter be called the "standard procedure."

In order to determine the possible effect of moisture or other components of the atmosphere that might have become adsorbed on the magnesium, a series of slurries was prepared by a method which will be called the "heating-flushing procedure." The magnesium powder was weighed into

a 1-liter resin flask equipped with a motor-driven, vacuum-sealed stirrer, gas-inlet tube, condenser, thermometer, addition funnel, and heating mantle. The powder was heated (320° to 350° F) and stirred while being alternately evacuated (to 5 to 7 mm pressure) and flushed with pure, dry helium in 2-hour cycles. This procedure required 6 to 8 hours. The powder was cooled to approximately 210° F under helium, and the n-decane (with additive, if any) was added.

When the powder was to be treated with moisture or air prior to the addition of the n-decane, the heating-flushing procedure was followed by cooling overnight under a gentle current of dry helium. The next day, the powder was treated for 6 hours with helium bubbled through a saturated, aqueous solution of potassium acetate (for humidification) or with air passed successively through three columns containing anhydrous calcium sulfate, activated silica gel, and anhydrous magnesium perchlorate (for treatment with dry air). Then the n-decane was added.

Test Methods

Brookfield apparent viscosity. - The Brookfield apparent viscosity was a one-point measurement of apparent viscosity at a low rate of shear, made at $86^{\circ} \pm 2^{\circ}$ F with a model LVF Brookfield Synchro-lectric viscometer. A diagram of this instrument is shown in reference 8. The number 3 spindle at 12 rpm (estimated rate of shear, 10 sec^{-1}) was used for viscosities below 10,000 centipoises, and the number 4 spindle at 6 rpm (estimated rate of shear, 0.5 sec^{-1}) was used for higher viscosities. The measurements were made in the same pint cans in which the slurries were prepared. The slurry was first stirred vigorously with a motor-driven stirrer. The viscometer spindle was then immersed to the mark and permitted to rotate for 30 ± 1 seconds before the reading was taken. The Brookfield apparent viscosity reported was the average of two or more determinations on the same sample.

Duplicate determinations almost always agreed within 10 percent of the average. The principal exceptions occurred when slurries were so thick and flowed so poorly that the rotating spindle alternately dragged against the slurry and broke loose. In such cases the indicating needle fluctuated between maximum and minimum readings. The range of such readings has been recorded in the tables and is shown in the graphs by a line parallel to the viscosity axis.

The accuracy of the Brookfield viscometer was within 5 percent over the range of viscosities measured when it was calibrated with Newtonian liquids of known viscosity.

Stormer flow curves. - Flow curves (plots of rate of rotation against load applied) were obtained for a limited number of slurries (after aging, as explained under DISCUSSION) at rates of shear up to about 1800 reciprocal seconds by means of a modified Stormer viscometer at approximately 86° F. This instrument and the method of obtaining data with it are described in reference 12. Because of the wide range of consistency of the slurries measured here, it was necessary to use another cylindrical cup and bob in addition to the one described in reference 12. The following are the dimensions of each cup and bob:

Cup and bob	Height of bob, in.	Diameter of bob, in.	Inside diameter of cup, in.
1	1.418	1.730	1.889
2	1.577	1.0225	1.1395

When a linear plot of rate of rotation against load was obtained, the plastic viscosity was calculated from the reciprocal of the slope, and the apparent yield value was obtained by determining the point where the extrapolated linear plot intersected the load axis. The following equations were used:

For cup and bob 1:

$$\mu = \frac{1.2}{\text{Slope of plot}}$$

$$f = 0.9 W_0$$

For cup and bob 2:

$$\mu = \frac{3.9}{\text{Slope of plot}}$$

$$f = 2.3 W_0$$

where

μ plastic viscosity, centipoises

f apparent yield value, dynes/sq cm

W_0 intercept of extrapolated plot with load axis, g

The accuracy of the Stormer viscometer appeared to be within 20 percent, insofar as could be judged from the following measurements on silicone oils:

Viscosity of silicone oil determined with Ostwald-type viscometer, centipoises	Temperature, °F	Viscosity measured with Stormer viscometer, centipoises	
		With cup and bob 1	With cup and bob 2
42	86	49	45
1000	77	970	1000

Severs flow curves. - Viscosity measurements were also made on the same slurries (after aging) with a Severs rheometer at 75° to 82° F and at rates of shear ranging approximately from 2000 to 24,000 reciprocal seconds. The construction, operation, and constants for this instrument in which the material is forced through an orifice under controlled pressure are discussed in reference 8. The data obtained were treated as described in reference 8 insofar as possible, but certain modifications were required as described in the discussion section of this report.

Sedimentation volume. - The extent of settling of the aged slurries was determined in ordinary 50-cubic-centimeter graduated cylinders (2.0 cm I.D.) at 86°±2° F. The cylinders were cleaned with hot chromic acid, rinsed thoroughly with distilled water, oven-dried, and then cooled just before use. Approximately 50 cubic centimeters of the well-mixed slurry was poured into the cylinder, which was then tightly sealed and set in a constant-temperature bath. As the magnesium settled to form a sharply demarcated layer, the volume of the settled portion was measured periodically. The sample was permitted to settle for 28 days or somewhat longer. For each reading, the sedimentation volume was calculated from the equation

$$\frac{\text{cc of sediment}}{\text{g of magnesium}} = \frac{v}{v_0 \rho c}$$

where

v volume of sediment, cc

v_0 volume of slurry, cc

ρ density of slurry, g/cc

c concentration of magnesium, percent by weight

The sedimentation volume was used as a measure of the extent of settling in lieu of the settling ratio and sedimentation ratio previously used (refs. 5 and 8) in a continuing effort to express the extent of settling while taking into account the concentration of magnesium.

Settling curves for slurries over a range of concentrations were drawn by plotting sedimentation volume against time (fig. 2). For the sake of convenience, however, with the exception of those shown on figure 2, only sedimentation volumes at 28 days are plotted and compared. The reproducibility of the sedimentation volume was good, as can be judged from the results for duplicate samples shown in figure 2.

Since the geometry of the container is such an important factor in sedimentation, these results were largely empirical and were therefore suitable only for comparing one slurry with another.

RESULTS AND DISCUSSION

Effect of aging. - Slurries containing additives that were surface-active almost always showed an appreciable change of Brookfield apparent viscosity with age. (Additives are considered to be surface-active when they are effective in reducing the viscosity of the slurry.) The change observed was dependent on the concentration of additive, as shown in a typical set of plots in figure 3. The viscosity of the slurry containing 0.10 percent lecithin (Al18) decreased exponentially with time, leveled off, and then began to increase. The behavior of the sample containing 0.50 percent lecithin was erratic, while the viscosity of the slurries containing 1.00, 2.00, and 4.00 percent lecithin decreased exponentially with time. The measurements on the last three slurries were discontinued because further decreases in viscosity became too small to be measured precisely with the Brookfield viscometer.

While the variations of viscosity with age shown in figure 3 are typical, deviations from this pattern were sometimes found when an additive less effective than lecithin was used to reduce the viscosity of the slurry. Examples of such behavior can be seen in figure 4 where the effect of aging on the viscosity of slurries containing *n*-octadecylamine (Al24) is indicated. With 1.00 and 2.00 percent Al24, the viscosity increased before it began to decrease. In fact, with 2.00 percent Al24, the initial increase was considerably greater than the subsequent decrease. With 0.50 percent Al24, the viscosity reached an essentially constant value after the exponential decrease.

The decreases in viscosity shown in figure 3 may be due to the time required for the molecules of the additive to move to the particle surfaces and become oriented at the interface. For example, when other investigators found that "some days" were required for the interfacial

tension between water and hexane solutions of surface-active additives to decrease to an equilibrium value (ref. 13), it was suggested that this occurred because the additive had become associated and that the rate-controlling process of dissociation had to precede adsorption (ref. 14).

The eventual increase in viscosity of the slurry containing 0.10 percent lecithin may have been caused by increased flocculation resulting from repeated incorporation of atmospheric moisture each time the slurry was stirred for viscosity measurements. This is substantiated later by data obtained with the heating-flushing procedure. At 1.00 percent or more lecithin, the concentration may have been sufficient to overcome this atmospheric moisture, while the erratic behavior at 0.50 percent may represent an intermediate condition.

Because of the effect of time, the final viscosity of a slurry was recorded and other measurements were made only after the slurry had been aged at room temperature. The slurry was aged until its Brookfield apparent viscosity had become essentially constant or had just begun to rise again after passing through a minimum.

Effect of magnesium from different drums. - Slurries of otherwise identical composition prepared with magnesium from the three different drums showed a large difference in Brookfield apparent viscosity. The slurries made with magnesium C had the highest viscosity, those made with magnesium B were intermediate, and the ones made with magnesium A had the lowest viscosity. In figure 5, this variation can be seen to hold, within experimental error, over the entire concentration range examined of the additive cetyl alcohol (A105). Similar differences were obtained with other additives. Because of these differences, the drum of magnesium used will be specified when data are presented.

Since the analyses of magnesium A and magnesium C are so similar (table I), it is presumed that the difference between them lies in the particle-size distribution of the fraction below 8.5 microns, or in the surface characteristics of the powders. In this connection, it was found that in dilute suspension in *n*-decane containing a surface-active additive, magnesium C appeared to settle more slowly than magnesium A.

These results serve to demonstrate the importance of the characteristics of the magnesium as a factor controlling the behavior of the slurry.

Effect of concentration of additive on apparent viscosity. - In order to determine how the Brookfield apparent viscosity of slurries varied with the concentration of additive, the Brookfield apparent viscosity was plotted against the concentration of several additives. It was necessary to express the concentration in percent by weight rather than on a molar basis because the molecular weights of many of the

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additives were unavailable or were known only approximately. Furthermore, when the molecules of additive are complex and have several polar groups, it is difficult to ascertain which of the polar groups participate in the adsorption process (see summary of literature in appendix B for relation between adsorption and surface activity). Comparison of additives on a weight basis is also desirable because it is necessary to keep the weight proportion of nonhydrocarbon compounds in the slurry to a minimum in order to obtain the maximum heat of combustion per unit weight of fuel.

Two types of viscosity-concentration curve were obtained. In one type, shown for three additives in figure 6(a), the viscosity decreased very rapidly with small increments of additive, and asymptotically approached a minimum viscosity. Essentially the maximum effect was achieved with 1.00 percent additive, although concentrations as high as 4.00 percent were tested. The additives which gave this type of curve were lecithin (Al18), polyoxyethylene sorbitol tetraoleate (Al32), and sodium dioctyl sulfosuccinate (Al36), which have bulky branched or ring structures with several polar groups per molecule.

In the other type of curve (fig. 6(b)), there was the same rapid decrease of viscosity with initial increments of surface-active additive, but a minimum was reached, followed by an increase in the viscosity as the concentration was further increased. The additives giving this type of curve were stearic acid (Al39), oleic acid (Al27), cetyl alcohol (Al05), and octadecylamine (Al24), all of which have slender straight-chain structures with only one polar group per molecule. The minimum in viscosity occurred in the neighborhood of 0.5 percent, except for cetyl alcohol, which gave a minimum near 1 percent. While the viscosity increase beyond the minimum was large with oleic acid or stearic acid, it was only moderate and of no practical consequence with either octadecylamine or cetyl alcohol.

Since it was necessary to evaluate the great majority of the additives at only one concentration so as to facilitate the work, 1.00 percent by weight was chosen as a basis for comparison as a result of the findings discussed previously. An exception was made in the case of three carboxylic acids, which were evaluated at 0.50 percent, because oleic and stearic acids gave a much lower viscosity at this concentration.

The viscosities of 4.00-percent solutions of Al05, Al18, Al27, and Al36 in *n*-decane were measured in order to determine whether the viscosity increases of the slurries beyond the minimums could be accounted for by the increased viscosity of the medium resulting from the additive. The results in table IV show that the viscosity of the solution never exceeded that of pure *n*-decane by more than 10 percent. Since empirical equations for the viscosity of suspensions indicate that the viscosity of a suspension is directly proportional to the viscosity of the medium

(e.g., refs. 15 and 16), the increase in the viscosity of the medium should not have been sufficient to cause the observed increase in the viscosity of the slurry.

A possible explanation for the minimums in figure 6(b) is the formation of a partial second adsorbed layer of additive with the polar group oriented toward the hydrocarbon medium so as to cause some reflocculation. The much greater increase in viscosity beyond the minimum when oleic acid was used may have resulted largely from the formation of magnesium oleate, which could form a gel with the *n*-decane. When a strip of magnesium which had been scoured under hydrocarbon was immersed in a 4.0-percent solution of oleic acid in *n*-decane for a week, a white sediment formed. Spectrographic analysis of this sediment showed that it contained magnesium. A parallel test without the oleic acid gave no sediment, and the liquid contained no magnesium.

Effect of concentration of additive on Stormer flow curves. - The effect of the concentration of two of the additives on the rheology of the slurry was determined by obtaining flow curves with a Stormer viscometer. Figure 7 shows Stormer viscometer flow curves for slurries containing various concentrations of cetyl alcohol (Al05). An analogous set of curves (not shown) was obtained with various concentrations of polyoxyethylene sorbitol tetraoleate (Al32) as the additive.

Pseudoplastic slurries were obtained with zero and 0.16 percent cetyl alcohol (fig. 7(a)). The small displacement (toward higher rates of rotation) of the down curve from the up curve indicates a small amount of thixotropy. The pseudoplastic behavior may have been due to the presence of clumps of particles that became oriented when the slurry was under shear in the viscometer. These clumps will be referred to as aggregates.

When 0.79 percent cetyl alcohol was used, the shape of the flow curve (fig. 7(b)) resembled more closely that for a plastic material, indicating that most or all of the aggregates had been broken up by the additive, and that the slurry was still flocculated. The down curve was still displaced slightly, but toward lower rates of rotation. An apparent yield value and plastic viscosity could be estimated from this flow curve. When 1.75 and 4.00 percent cetyl alcohol were used, the flow curves were definitely those of a plastic (flocculated) material, with the up and down curves coinciding. In figures 7(c) and (d), the flow curve has a sharp bend. Since a bend had been observed in the flow curve for 60.6 percent glycerol in water, which is known to be Newtonian, it is believed that the flow of the slurry in the viscometer became turbulent at rates of rotation above that corresponding to the bend. The lower portion of the flow curve was therefore used to calculate plastic viscosity and apparent yield value.

Examination of table V shows that the plastic viscosity of slurries containing Al32 decreased with increasing concentration of additive over the range examined. When cetyl alcohol (Al05) was used, the plastic viscosity decreased to about 13 centipoises and then increased somewhat as the concentration was increased. The change of apparent yield value with concentration of surface-active additive showed the same trend as the change of plastic viscosity with concentration of additive (table V). When cetyl alcohol was used, the minimum apparent yield value was about 22 dynes per square centimeter.

Effect of additive concentration on Severs flow curves. - The shape and relative position of the Severs flow curves also varied with the concentration of additive. This variation is shown in figure 8 for the same slurries containing cetyl alcohol which have been discussed in connection with the Stormer viscometer. The slopes of these linear (within experimental error) plots increased with increasing cetyl alcohol concentration up to 1.75 percent and then decreased. When the additive was Al32, the plots were somewhat curved (not shown) and became increasingly steep as the concentration of additive increased to 2.00 percent, but without reaching a maximum steepness. None of the Severs flow curves for slurries passed through the origin.

When possible, the residual viscosity of the slurries was determined as was done in reference 8 by plotting reciprocal of rate of shear against Severs apparent viscosity and extrapolating to the apparent viscosity at an infinite rate of shear (e.g., curve for 0.16 percent cetyl alcohol, fig. 9). However, this was not possible for the more fluid slurries (see curves for 0.79, 1.75, and 4.00 percent cetyl alcohol, fig. 9) because the plots were vertical lines at the lower rates of shear and then assumed a negative slope at the higher rates of shear. Since a Newtonian liquid consisting of 60.6 percent glycerol in water gave a similar plot (inset, fig. 9), the bend is probably the result of some characteristic of the instrument, such as turbulent flow at the higher shear rates. The vertical portions of the plots in figure 9 therefore represent the minimum Severs apparent viscosity of the slurries in laminar flow, and these are the values reported. For the glycerol solution, the vertical portion of the plot falls at 8.5 centipoises at 81° F, which agrees with 8.6 centipoises determined with an Ostwald type of viscometer. The Severs apparent viscosities listed in table V are the residual or minimum viscosities.

Comparison of viscosity measurements. - Table V contains measurements made with the Brookfield, Severs, and Stormer viscometers on the same slurries containing various concentrations of Al05 and Al32. The Severs viscosities and the Stormer plastic viscosities are of the same order of magnitude. The Brookfield apparent viscosities are 1 to 2 orders of magnitude higher than the Severs or Stormer values, presumably because of the low rate of shear at which the Brookfield instrument was used.

Although the viscosity measurements obtained with all three viscometers follow similar trends (table V), a minimum viscosity was found at 0.79 percent AlO₅ when the Brookfield viscometer was used, while the minimum appeared at 1.75 percent AlO₅ when either the Stormer or Severs viscometer was used. This observation again indicates a greater similarity between the results obtained with the Stormer and Severs instruments when compared with those obtained with the Brookfield viscometer.

3451 Effect of heating-flushing procedure. - The slurries which have heretofore been discussed were made by the standard procedure with magnesium that had been exposed to the atmosphere. By applying the heating-flushing procedure (heating the magnesium in a flask while alternately evacuating it and flushing it with dry helium, and then adding the n-decane to the magnesium in the flask), a slurry with a lower viscosity was obtained. Such slurries also showed aging characteristics which were different from those of slurries prepared by the standard procedure.

The Brookfield apparent viscosity of slurries made by the standard procedure and by the heating-flushing procedure and containing zero, 0.1, and 2.0 percent lecithin (All8) are compared in table VI. This table shows that the heating-flushing procedure gave a slurry with a lower Brookfield apparent viscosity than the standard procedure, the difference being greatest when no surface-active additive was present. While the heating-flushing procedure alone apparently partially prevented flocculation of the slurry, it was not as effective as 2.0 percent lecithin in the standard procedure. For practical purposes, the use of surface-active additives to reduce the viscosity of a slurry is much more economical and convenient than the heating-flushing procedure.

It is believed that the heating-flushing procedure removed some of the adsorbed polar substance, such as moisture or some other component of the air, which promoted flocculation. The heated-flushed magnesium was treated in separate experiments with humidified helium and with dried air prior to the addition of the n-decane in an attempt to test the hypothesis and to find out whether moisture or some other component of the air was involved. From the data in table VI, it is evident that moisture was the most important factor, since the treatment with moist helium gave a slurry with approximately the same viscosity as the one made with the standard procedure. However, air may also have been a factor, since flushing with dry air gave a slurry of intermediate viscosity. Although the air used was believed to have been dry, it may not have been freed of every trace of moisture by the drying procedure used.

The effect of aging was also different when the heating-flushing procedure rather than the standard procedure was employed, as shown by a comparison of figures 3 and 10. Figure 10 consists of plots of Brookfield apparent viscosity against aging time for slurries made by the heating-flushing procedure and containing zero to 2.0 percent lecithin.

With no lecithin, the viscosity began to rise rapidly from the time the slurry was prepared. With 0.1 percent lecithin, the viscosity dropped somewhat at first, then rose considerably. It is believed that the increase in viscosity of these two slurries resulted from restoration of part of the moisture as a result of the vigorous stirring with exposure to the atmosphere preceding each measurement of viscosity. This increase in viscosity appears to be similar to the one shown in figure 3 for a slurry containing 0.10 percent lecithin, except that it occurred sooner. With 2.0 percent lecithin, the viscosity changed very little with age instead of decreasing markedly (see fig. 3). These differences in the effect of aging would be the logical result of removing the moisture initially present in the powder. Here, as in the case of the standard procedure, a sufficient concentration of lecithin prevented an ultimate increase in viscosity.

Effect of temperature. - When the Brookfield apparent viscosities of three slurries were measured over a range of temperatures (approximately from 0° to 140° F), the viscosity decreased as the temperature was increased (fig. 11). The slurry containing 1.00 percent Al32 was the most fluid. The other two, one of which contained 0.20 percent Al32 and the other 1.00 percent Al07, were considerably thicker. The viscosity-temperature plots for the two thicker slurries were approximately as steep as the plot for pure n-decane (fig. 11), while that of the thin slurry had a much smaller slope. Over the temperature range examined, the viscosities of the two thicker slurries and of pure n-decane changed approximately 300 percent. The viscosity change for the thinner slurry was much less, about 30 percent. This difference cannot be attributed to a difference in the effect of temperature on the viscosities of pure n-decane and n-decane containing 1.00 percent Al32, since the temperature-viscosity plots for the two almost coincide (fig. 11). (Although a 50-percent magnesium slurry containing 1.00 percent of additive based on the weight of slurry would contain 2.00 percent based on the weight of hydrocarbon, a 1.0 percent solution of Al32 in n-decane was used for comparison because the magnesium would probably have adsorbed about half the additive from the solution.) The difference between the slopes of the temperature - apparent viscosity plots of these slurries may therefore be the result of differences in the effect of temperature on the interaction between particles of magnesium or between the particles of magnesium and the hydrocarbon medium.

Relation of apparent viscosity to sedimentation volume. - The extent of settling of the slurry in 28 days, as measured by the sedimentation volume, decreased with increasing Brookfield apparent viscosity, as shown in figure 12. In this figure, in which the sedimentation volumes of all the slurries prepared with magnesium A and magnesium C by the standard procedure are plotted against Brookfield apparent viscosity, the points are scattered, but the trend of the points is quite definite. An area has been shaded to indicate a band in which a great majority of the

points fell. A Brookfield apparent viscosity of 2500 centipoises or more was required to obtain little or no settling. (A sedimentation volume of 1.9 to 2.0 indicates that no settling occurred. This value varies slightly with the density of the slurry.) The data in figure 12 indicate that all the slurries which were very fluid settled a great deal. While it appears that not all additives will produce the same sedimentation volume at a given viscosity, figure 12 suggests that a 50-percent slurry of this type of magnesium in *n*-decane containing nothing else but a small concentration of surface-active additive must have a high apparent viscosity to prevent settling during storage. This figure also demonstrates that fairly fluid slurries (approximately 2000 centipoises Brookfield apparent viscosity) which settle only moderately (sedimentation volume of 1.3 to 1.7 cc/g) can be prepared. This condition can readily be achieved by judicious choice of the additive.

A smaller sedimentation volume was correlated with a harder cake of sediment. In the most fluid slurries, which had the smallest sedimentation volumes, the magnesium settled into a very hard cake which made remixing very difficult. When the sedimentation volume was high, remixing was very easy.

Comparison of additives. - The additives tested covered a wide range of surface activity, as indicated by the range of Brookfield apparent viscosities obtained by using 1.00 percent of the various additives (except Al08, Al27, and Al39, all 0.50 percent). The additives are listed in order of increasing viscosities of slurry in table VIII, in which the data obtained with magnesiums A, B, and C are listed separately.

The data in table VIII for the three powders have been grouped somewhat arbitrarily into three ranges of viscosity as follows:

Range	Brookfield apparent viscosity, centipoises		
	Magnesium A	Magnesium B	Magnesium C
1	<300	<500	<700
2	300-3500	500-3500	700-3500
3	>3500	>3500	>3500

In table IX, these viscosity ranges are correlated with the structure of the polar groups in the molecules of additive. It can be seen that all the additives that gave viscosities in range (1) had a hydroxyl group together with an ester, metal salt, or polyoxyethylene group. Of 13 such compounds tested, 10 gave slurries in range (1), and the other 3 fell into range (2). If only one of these groups was present, or if a primary amine was present, the viscosity fell into range (2) (with two

exceptions out of 26 additives). If none of these groups was present (with only one exception) the viscosity fell into range (3), that is, the additive had little or no effect in reducing the viscosity of the slurry.

Evidently the common classification of surface-active additives as ionic or nonionic is not useful here. Thus, among the additives giving slurries in viscosity range (1) there are A101, A118, and A134, which are considered ionic, and seven others that are considered nonionic.

Within the limits investigated, molecular weight had little effect (e.g., cetyl alcohol (A105) and n-octyl alcohol (A125) both in range (2) and A109 and A132 in range (1)). This lack of effect is further illustrated by a comparison of slurry viscosity with the number of millimoles of additive per 400 grams of slurry (table VIII). There appears to be no correlation between these values, even when an attempt is made to take the number of polar groups per molecule into consideration.

When the molecule contained only one polar group, a bulky molecule appeared to be less effective than a slender one. For example, dehydroabietyl amine (A106) gave a higher viscosity than n-octadecylamine (A124) (see table VIII). However, the lowest viscosities were obtained with some of the bulkiest molecules which had multiple polar groups. The wide range of Brookfield apparent viscosities which was obtained indicates that there is considerable latitude in the choice of additives. If the most fluid slurry containing 50 percent magnesium of the type used in this investigation were desired, about 1 percent of A110, A132, A118, or a similar additive having a hydroxyl group together with an ester, metal salt, or polyoxyethylene group in the molecule would give the desired viscosity. Any one of a number of other additives (see range (2) in table VIII) would give somewhat more viscous slurries. However, all these slurries would settle a great deal, and the settled cake would be stiff and difficult to remix. A moderately viscous slurry could be prepared with about 0.2 percent of an additive like A110, A132, or A118, or with approximately 1 percent of an additive similar to A116 or A136. Such slurries would settle less and be much easier to remix.

Proposed mechanism for effect of surface-active additives. - The following mechanism is proposed on the basis of the experimental evidence:

A slurry prepared by the standard procedure without an additive is both aggregated and flocculated as the result of adsorbed polar material (most probably moisture, as shown by the results obtained with the heating-flushing procedure). The addition of a sufficient quantity of a surface-active additive simultaneously breaks up the aggregates and partially deflocculates the suspension. The deflocculation is not complete, since the yield value never becomes zero. The observed changes in this process are a marked decrease in apparent viscosity at a low

rate of shear, a transition from a pseudoplastic to a plastic material, a decrease in plastic viscosity and apparent yield value, a decreased sedimentation volume, and a stiffer sediment.

The experimental evidence indicates that one function of the surface-active additive is to counteract the flocculating and aggregating effect of adsorbed moisture. This process requires an appreciable time to reach equilibrium unless most of the moisture has been removed previously by the heating-flushing procedure. If the quantity of additive is insufficient, atmospheric moisture repeatedly stirred into the slurry will be more than can be counteracted by the additive and will increase the degree of flocculation by being adsorbed on some of the available surface of the particles. If sufficient additive is present, atmospheric moisture has no effect, either because there is no surface available for it on the particles or because it becomes associated with the additive present in the bulk of the medium.

Other investigators have found that the effect of surface-active additives is the result of their adsorption on the surface of the particles (see summary of the literature, appendix B). The adsorption involves an interaction between the surface and the polar groups in the molecule of additive. Since magnesium reacts so readily with oxygen, and the powder was found to contain 8 to 12 percent magnesium oxide, it can be presumed that the surfaces of the particles are largely or entirely coated with magnesium oxide, which may be hydrated or carry adsorbed moisture. The mechanism of adsorption could involve hydrogen bonding between the magnesium oxide or moisture and the amine, carboxyl, ester, ether, hydroxyl, or metal salt groups of the additive. A similar mechanism has been hypothesized in reference 17 for adsorption on hydrated silica gel. Other possible mechanisms involve chemical reaction, as has been demonstrated by the action of oleic acid on magnesium ribbon, or the coordination of an oxygen atom of the additive to the magnesium atom of the oxide. This arrangement is analogous to the coordinated structure suggested for a Grignard reagent - ether complex in reference 18. If there is any exposed metallic magnesium on the surfaces of the particles, adsorption of molecules containing carboxyl groups could occur by chemical reaction. In the event the active group were an ester, hydroxyl, or amine, the dipole involved is generally believed to interact with the metal.

The occurrence of one or more of these mechanisms serves to explain why a molecule of additive which has both hydroxyl and polyoxyethylene, ester, or metal salt groups is particularly effective in reducing the viscosity of the slurry. A multiplicity of such groups in the molecule of additive provides several points at which interaction between the molecule and the particle can occur. This multiplicity of polar groups enhances the attractive force between the molecule and the particle and probably causes the additive molecule to occupy effectively a larger surface than

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CR-3

would a slender molecule with only one point of attachment. In this way, a multiplicity of polar groups promotes a greater degree of deflocculation.

SUMMARY OF RESULTS

In an investigation of the effect of additives on the physical properties of slurries containing 50 percent by weight of 1.5-micron average equivalent spherical diameter magnesium in purified n-decane, the following results were obtained:

1. In the absence of a surface-active additive, the slurries exhibited (a) the high apparent viscosity, (b) the small extent of settling, and (c) the soft sediment predicted for a concentrated suspension of polar particles in a nonpolar medium. These slurries were pseudoplastic, thixotropic materials.

2. As the slurries were made increasingly fluid by the incorporation of a surface-active additive, the following effects were observed:

(a) The extent of settling in 28 days became greater. Generally, only when the Brookfield apparent viscosity exceeded 2500 centipoises was there little or no settling.

(b) In the most fluid slurries, the magnesium settled into a hard cake which was very difficult to remix.

(c) In the limited number of cases tested, the slurries became less pseudoplastic; and when they were made sufficiently fluid, they became plastic, nonthixotropic materials.

3. Heteropolar compounds containing one or more hydroxyl, amine, amine salt, carboxyl, ester, or metal salt groups were the additives that were appreciably surface-active; that is, they appreciably reduced the Brookfield apparent viscosity of the slurries.

4. The additives that were the most surface-active had one or more hydroxyl groups together with polyoxyethylene, ester, or metal salt groups in the molecule.

5. When the surface-active additive was a bulky molecule with a branched or ring structure and had a multiplicity of polar groups, the Brookfield apparent viscosity asymptotically approached a minimum as the concentration of additive was increased. The decrease in viscosity beyond a concentration of 1 percent of additive, based on the weight of slurry, was minor.

6. When the surface-active additive was a slender molecule with only one polar group, the Brookfield apparent viscosity decreased as the concentration of additive was increased until it reached a minimum in the neighborhood of 0.5 to 1.0 percent additive by weight. Except in the case of carboxylic acids, the viscosity increase beyond the minimum was minor.

7. The Brookfield apparent viscosity of a slurry containing a surface-active additive changed with time. The usual effect was for the viscosity to decrease and level off. If the concentration of additive was in the neighborhood of 0.1 percent, the viscosity decreased at first, and then increased on further aging. Some additives caused the viscosity to rise initially before decreasing.

8. The removal of part of the moisture (or possibly other components of the air) from the magnesium powder before the n-decane was added caused a decrease in the Brookfield apparent viscosity.

9. Slurries of the same composition, but made with magnesium powder taken from three different drums, had markedly different Brookfield apparent viscosities even though the available test data indicated that the powders should be similar.

10. When the effect of temperature was evaluated for three slurries, the change in Brookfield apparent viscosity as the temperature was decreased from 140° to 0° F appeared to depend on the concentration and composition of the surface-active additive.

11. The Stormer plastic viscosity and apparent yield value decreased continuously with increasing concentration of polyoxyethylene sorbitol tetraoleate (A132), but decreased to a minimum and then increased somewhat with increasing concentration of cetyl alcohol (A105) (within the concentration ranges examined).

12. In the limited number of comparisons which were made, the Severs minimum or residual viscosity and the Stormer plastic viscosity were of the same order of magnitude, and both were much lower than the Brookfield apparent viscosity.

CONCLUDING REMARKS

While the data that have been reported were obtained from slurries consisting only of 50 percent of a specific grade of 1.5-micron magnesium, n-decane, and a small concentration of certain additives, the results obtained should be applicable to modifications of this formula if the properties of each component are taken into account. For example, if, all other conditions being equal, a more dense hydrocarbon were used

instead of n-decane, the higher density of the liquid would result in a thicker slurry because of the smaller volume fraction of liquid. Also, the presence of polar impurities in a hydrocarbon such as MIL-F-5624A, grade JP-4, could influence the viscosity. If the particle-size distribution, average particle size, or particle shape of the magnesium were appreciably different, the flow properties and settling behavior would be affected. With more finely divided magnesium, the viscosities obtained would be higher, and therefore more additive or a more powerful additive would be required. If the magnesium were sufficiently finely divided, even 1 percent or more of an additive like ALLO might not reduce the viscosity so much that settling would be a problem. These factors, which must be considered in the formulation of slurries, are also some of those that must be controlled in order to obtain uniform batches of slurry once a formulation has been selected.

The relation between Brookfield apparent viscosity and the sedimentation volume and hardness of the cake of sediment that has been found in this investigation indicates that the viscosity of the slurry should be maintained at as high a value as is consistent with proper pumping, atomization, and other mechanical procedures, in order to minimize settling and formation of a hard sediment. For the slurries which have been discussed, this can be done not only by choice of the type and concentration of surface-active additive but also by incorporation of a suitable thickening agent such as petrolatum. For slurries of much finer magnesium, a thickener may be undesirable because the viscosity would be sufficiently high without it. The possible effect of thickeners and surface-active additives on the combustion properties of the slurries is an important consideration.

While the mechanisms which have been proposed to explain the effect of surface-active additives on slurries are not of immediate practical importance, they may eventually lead to a better understanding of the behavior of slurries. For the present, the finding of a correlation between the structure of the additive and the viscosity of the slurry should be useful as a guide in predicting the behavior of other additives. The viscosity of the slurries discussed in this report can be controlled over a wide range by the use of the proper concentration of one of the 50 additives that have been evaluated.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 1, 1954

APPENDIX A

GLOSSARY

Aggregation - The condition which exists when there are present in suspension clumps (aggregates) of particles that can be broken up permanently either by the application of mechanical forces, or by the incorporation of suitable surface-active additives.

Flocculation - The condition which exists when particles in suspension come together to form clusters. These clusters can be broken up permanently only by the incorporation of suitable surface-active additives.

Flow curve - A plot of rate of shear (ordinate) against shearing stress (abscissa). For a Stormer viscometer, the rate of shear may be expressed as the rate of rotation of the bob, and the shearing stress may be expressed as the load applied. When the plot is obtained by measuring the rate of shear at successively increasing shearing stresses, it is called an up curve. For decreasing rates of shear, it is called a down curve.

Newtonian - A material for which the flow curve is a straight line passing through the origin.

Plastic - A non-Newtonian material for which the flow curve is a straight line (except possibly at very low rates of shear) which, when extrapolated, intersects the shearing stress axis at a positive value.

Pseudoplastic - A non-Newtonian material for which the flow curve (both up curve and down curve) starts at the origin, is nonlinear, and convex toward the shearing stress axis.

Thixotropy - A condition in which the structure of a suspension is destroyed by agitation and is rebuilt upon rest. It is evidenced by a flow curve in which, for a given shearing stress, the rate of shear is higher on the down curve than on the up curve.

Viscosity, apparent - The reciprocal of the slope of a line drawn from the origin to any point on the flow curve.

Viscosity, plastic - The reciprocal of the slope of the linear flow curve exhibited by a plastic material.

Viscosity, residual - When the plot of reciprocal of rate of shear against apparent viscosity is a straight line, the value of the intercept of the extrapolation of this line with the apparent viscosity axis is the residual viscosity.

Yield value - The value of the intercept of the extrapolated linear flow curve of a plastic material with the shearing stress axis.

APPENDIX B

SUMMARY OF PERTINENT LITERATURE CONCERNING SUSPENSIONS
AND SURFACE-ACTIVE ADDITIVES

3451 While the literature contains very little mention of the rheology and settling behavior of concentrated suspensions of metals in hydrocarbons that have not been modified with thickening agents, considerable experimental data and some theories regarding concentrated suspensions of a number of other solids in hydrocarbons have been published. Some of the theories represent conflicting concepts. The summary which follows includes the theories that appear to be most firmly based on experimental evidence.

Properties of flocculated suspensions. - When a high concentration of polar solid is mixed with a nonpolar medium, a flocculated dispersion results (refs. 19 and 20). That is, the particles tend to form a sort of continuous network of clusters which, when under shear, is continuously broken and reformed (ref. 20). Flocculation in a concentrated suspension is manifested by:

- (1) A plastic flow curve (refs. 20 and 21); it follows that the apparent viscosity will be higher than the plastic viscosity
- (2) "Collective" settling; that is, the solid material settles in bulk, with a sharp line of demarcation between the settled portion and the supernatant liquid (ref. 22)
- (3) A relatively high settled volume (ref. 23) and relatively soft packing of the settled portion (ref. 24)

It has been stated that the last two phenomena (item (3)) are the result of a scaffolding of the flocculated structure, which causes the particles to settle as a group with a larger volume of interstices than would exist if each particle settled individually (refs. 23 and 25).

Mechanism of flocculation. - Flocculation has been attributed to contact between particles, either directly or by the "bridging" action of adsorbed polar substances, such as water, which are only slightly soluble in the medium. The process of flocculation occurs spontaneously as a means of decreasing the interfacial area and thereby the interfacial energy (ref. 19). Suspensions are not all flocculated to the same extent, the degree of flocculation of a given system being related to the interfacial tension between the medium and the surface of the particle itself or any adsorbed film on the particle (ref. 21).

Adsorption of a compound with a polar group is apparently a primary factor in the behavior of suspensions like those of magnesium in n-decane. Such compounds, for example, water, alcohols, or fatty acids, are attracted to the interface between the particle and the hydrocarbon and are adsorbed on the surface of the particle. Adsorption has been established in numerous investigations, such as those described in references 26 to 28 where it was shown that crushed glass partially removes acids, alcohols, and esters from hydrocarbon solutions. Similarly, magnesium oxide has been found to remove hexadecanol from a hydrocarbon solution (ref. 29).

When the adsorbed polar molecules have an insignificant nonpolar portion, as in water or ethylene glycol, they cause flocculation by bridging, as previously mentioned. The degree of flocculation depends on the nature of the adsorbed molecules. For example, when a series of liquids immiscible with hydrocarbons was added to titanium dioxide - hydrocarbon suspensions, the yield value increased in proportion to the interfacial tension between the added liquid and the hydrocarbon (ref. 30). It has also been shown that the sediment volume of titanium dioxide in benzene is increased even by traces of moisture, and that this moisture can be removed and kept out only by the most rigorous dehydration in a sealed system (refs. 31 and 32).

Mechanism of deflocculation. - When the adsorbed molecules are heteropolar, such as oleic acid, they decrease the degree of flocculation. Additives that behave in this way are commonly called surface-active agents. It is explained that the molecules of additive become oriented with the polar portion toward the particle surface and the nonpolar portion toward the hydrocarbon, effectively reducing the interfacial tension between the particle and the medium (ref. 33). Furthermore, if the forces between the particle and the polar portion of the additive molecule are sufficiently strong, a previously adsorbed material, such as water, will be displaced, and this also serves to decrease flocculation (ref. 34). Some investigators have stated that for a given additive the maximum deflocculation occurs when the quantity added to the suspension is just enough so that a close-packed monomolecular layer of additive is formed on the particle surface, and that an excess will cause partial reflocculation (ref. 33). Others have said that more than enough additive to give a monomolecular film is required to give maximum deflocculation (ref. 35).

APPENDIX C

SLURRIES CONTAINING COMBINATION OF TWO

SURFACE-ACTIVE ADDITIVES

Two sets of slurries were prepared with combinations of two additives. The Brookfield apparent viscosities of these slurries are listed in table X. These data indicate that the use of 1.00 percent of a combination of Al32 and Al24 offers no appreciable advantage over 1.00 percent Al32 alone insofar as Brookfield apparent viscosity is concerned. A combination of 0.50 percent Al32 and 0.50 percent Al36 likewise offers no advantage over 1.00 percent Al32. It is of interest, however, that when a combination of Al32 and Al36 was used, the viscosity reached a minimum at 0.50 percent total additive, even though neither additive alone showed a minimum in the viscosity - concentration curves in figure 6(a).

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TABLE I. - ANALYSIS OF MAGNESIUM POWDER

Property	Magnesium	Magnesium	Magnesium
	A	B	C
Assay, percent magnesium ^a	88	86	92
Magnesium oxide ^b , percent	12	14	8
Mean equivalent spherical particle diameter ^c , microns	1.5	1.4	1.5
Particle size distribution ^d , percent by weight			
Below 8.5 microns	61	69	60
8.5 to 17 microns	34	14	30
17 to 26 microns	4	10	8
26 to 40 microns	<1	6	1
Over 40 microns	Trace	approx. 1	1
Density ^e , g/cc	1.75	--	1.76

^aBy evolution of hydrogen in acid and conversion of hydrogen to water.

^bBy difference.

^cBy air permeability with Fisher Sub-Sieve Sizer.

^dBy air elutriation with Roller particle-size analyzer. Dimension given is equivalent spherical particle diameter. The Roller analyzer was used although it is not recommended for particles less than 4 microns in diameter.

^eBy volume displacement of n-decane.

TABLE II. - PHYSICAL PROPERTIES OF n-DECANE

Property	Found	Literature
Melting point, °F	-21.39	^a -21.390
Boiling point at 760 mm, °F	345.306	^b 345.421
Density at 68° F, g/cc	0.72995	^b 0.73005
Refractive index, n_D , 68° F	1.41193	^b 1.41189
Interfacial tension against water at 81° F, dynes/cm	51.1	^c 50.9

^aFreezing point, ref. 36.

^bRef. 36.

^cInterpolated from data in ref. 37.

TABLE III. - COMPOSITION AND PHYSICAL PROPERTIES OF ADDITIVES^a

Additive	Composition	Polar groups in molecule of additive	Purity or active ingredient, percent	Approximate molecular weight	Density, g/cc or specific gravity (at approx. room temp.)
A100	Alkylaryl sulfonate, amine salt (details unavailable)	-----	90+	390	1.03
A101	Aluminum hydroxynaphthenate	Hydroxyl, metal salt of carboxylic acid	^b 50	---	.9
A102	Aluminum hydroxydi(2-ethylhexoate)	Hydroxyl, metal salt of carboxylic acid	Technical	330	Approx. 1
A103	Bisphenol-epichlorohydrin condensate	Hydroxyl, ether	Approx. 100	---	---
A104	n-Butyl ether	Ether	Technical	130	.77
A105	Cetyl alcohol	Hydroxyl	Technical	242	.80
A106	Dehydroabietyl amine	Primary amine	Technical	290	1.0
A107	Dehydroabietyl amine stearate	Primary amine salt	Technical	570	1.0
A108	2-Ethylhexoic acid	Carboxylic acid	Technical	144	.90
A109	Glycerol sorbitan laurate	Hydroxyl, ester, ether	97+	330	1.00-1.05
A110	Glyceryl monooleate	Hydroxyl, ester	Technical	358	.95
A111	n-Hexyl bromide	Halide	Technical	185	1.17
A112	Hydroxytitanium stearate	Hydroxyl, ester, -Ti-O-Ti-O-	Technical	---	1
A113	Iron naphthenate	Metal salt of carboxylic acid	^b 75	---	1.0
A114	Isopropoxytitanium stearate	Ester, -Ti-O-Ti-O-R	Technical	---	1
A115	Lauryl methacrylate-diethylaminoethyl methacrylate copolymer (details unavailable)	Ester, tertiary amine	^b 50	---	1
A116	Lead di(2-ethylhexoate)	Metal salt of carboxylic acid	^b 80	493	1.1
A117	Lead dimaphthenate	Metal salt of carboxylic acid	^b 65	---	1.2
A118	Lecithin (derived from soybeans)	Ester, hydroxyl, quaternary ammonium	^c 100	780	.73
A119	Linoleic acid, dimerized	Carboxylic acid	^d 100	600	.95
A120	α-Methylbenzyl diethanolamine	Hydroxyl, tertiary amine	Technical	208	1.03
A121	α-Methylbenzyl monoethanolamine	Hydroxyl, secondary amine	Technical	185	1.08
A122	Mixed tert-alkyl primary amines, C ₁₈ to C ₂₄	Primary amine	Approx. 90	269-353	.83
A123	Nitrobenzene	Nitro	Approx. 100	123	1.2
A124	n-Octadecylamine	Primary amine	^e 100	276	.8
A125	n-Octyl alcohol	Hydroxyl	Technical	130	.89
A126	Octylene glycol titanate	Hydroxyl, Ti-O-R	^b 50	---	1
A127	Oleic acid	Carboxylic acid	U.S.P. XII	282	.85
A128	Polyoxyethylene dodecyl alcohol, 2 moles ethylene oxide	Hydroxyl, polyoxyethylene	97+	270	1

^aData either supplied by manufacturer or estimated from other information.^bRemainder is mainly hydrocarbon solvent. Sufficient additive used in slurries to give desired concentration of active ingredient.^c65 Percent lecithins, 35 percent soybean oil.^d85 Percent dimer, 12 percent trimer, 3 percent monomer.^e83 Percent n-octadecylamine, 8 percent n-hexadecylamine, 1 percent n-octadecenylamine.

TABLE III. - Concluded. COMPOSITION AND PHYSICAL PROPERTIES OF ADDITIVES^a

Additive	Composition	Polar groups in molecule of additive	Purity or active ingredient, percent	Approximate molecular weight	Density, g/cc or specific gravity (at approx. room temp.)
A129	Polyoxyethylene dodecyl alcohol, 4 moles ethylene oxide	Hydroxyl, polyoxyethylene	97+	360	1
A130	Polyoxyethylene dodecyl alcohol, 8 moles ethylene oxide	Hydroxyl, polyoxyethylene	97+	540	1
A131	Polyoxyethylene dodecanethiol, 3.5 moles ethylene oxide	Hydroxyl, polyoxyethylene, thioether	Technical	360	1.0
A132	Polyoxyethylene sorbitol tetraoleate	Hydroxyl, polyoxyethylene, ester	97+	1500	0.92-0.98
A133	N,N' Polyoxyethylene hydrogenated tallow amide, 5 moles ethylene oxide	Polyoxyethylene, amide, hydroxyl	Technical	490	1.03
A134	N,N' Polyoxyethylene hydrogenated tallow amine, 5 moles ethylene oxide	Polyoxyethylene, tertiary amine, hydroxyl	Technical	470	.97
A135	Sodium alkylaryl sulfonate (details unavailable)	-----	-----	---	1
A136	Sodium dioctyl sulfosuccinate	Ester, metal salt	Approx. 100	444	.73
A137	Sorbitan monooleate	Hydroxyl, ester, ether	97+	430	1.00-1.05
A138	Soybean oil (fatty acid triglycerides)	Ester	Technical	885	.9
A139	Stearic acid	Carboxylic acid	U.S.P. XII	270	.85
A140	Stearylamine salt of stearyl-amido-phosphoric acid, monoalkyl ester	Ester, secondary amine, amine salt	Technical	650-750	.9
A141	Substituted oxazoline (details unavailable)	-----	-----	---	1
A142	Triethanolamine oleate	Ester, tertiary amine	Technical	941	.9
A143	Triethylene glycol dimethyl ether	Polyoxyethylene	Technical	178	.9
A144	Proprietary product. Composition unavailable	-----	b93	---	.88
A145	Proprietary product. Composition unavailable	-----	b77	---	.97
A146	Proprietary product. Composition unavailable	-----	Approx. 100	437	1
A147	Proprietary product. Composition unavailable	-----	-----	---	.99
A148	Proprietary product. Composition unavailable	-----	b40	---	.89
A149	Proprietary product. Composition unavailable	-----	b37	---	.91

^aData either supplied by manufacturer or estimated from other information.^bRemainder is mainly hydrocarbon solvent. Sufficient additive used in slurries to give desired concentration of active ingredient.^cProbably consists of approximately 60 percent stearic acid and 40 percent palmitic acid.

TABLE IV. - EFFECT OF ADDITIVES ON VISCOSITY OF n-DECANE

Additive	Additive concentration in <u>n</u> -decane, percent	^a Viscosity at 86° F, centipoises	Viscosity increase over pure <u>n</u> -decane, percent
None	0	0.798	
Al36	4.00	.860	7.8
Al18	4.00	.875	9.7
Al27	4.00	.878	10.0
Al05	4.00	.866	8.5

^aViscosity determined from measurements with Ostwald-type viscometer.

TABLE V. - MEASUREMENTS MADE WITH THREE DIFFERENT
VISCOMETERS ON SAME SLURRIES^a

Additive	Concentration of additive, percent by weight	Brookfield apparent viscosity at 86° ± 2° F, centipoises	Severs minimum or residual viscosity at 75° to 82° F, centipoises	Stormer plastic viscosity at approx. 86° F, centipoises	Stormer apparent yield value, dynes/cm ²
Al05	0.16	4120	27	(b)	(b)
	.79	600	11	^c 52	^c 55
	1.75	690	8	13	22
	4.00	750	11	14	31
Al32	0.10	2920	(b)	(b)	(b)
	.20	1150	31	^c 27	^c 69
	.50	280	7	8	10
	1.00	200	6	7	9

^aMagnesium A, standard procedure.

^bValue could not be determined because flow curve was not linear.

^cFlow curve had a loop (see fig. 7(b)). Value estimated from average of two slopes or two intercepts.

TABLE VI. - EFFECT OF STANDARD AND HEATING-FLUSHING PROCEDURES
ON BROOKFIELD APPARENT VISCOSITY OF MAGNESIUM SLURRIES^a

Lecithin (All8) added, percent	Brookfield apparent viscosity at $86^{\circ} \pm 2^{\circ}$ F, centipoises	
	Standard procedure	Heating- flushing procedure
None	100,000-30,000	2400
0.1	2800	1970
2.0	220	150
None	100,000-30,000	2400
None ^b		100,000-20,000
None ^c		30,000-10,000

^aSlurries containing 50 percent Magnesium A.

^bMagnesium treated with humidified helium in addition to heating-flushing procedure.

^cMagnesium treated with dried air in addition to heating-flushing procedure.

TABLE VII. - SEDIMENTATION VOLUMES AND BROOKFIELD APPARENT VISCOSITIES

OF SLURRIES MADE WITH MAGNESIUM A AND MAGNESIUM C^a

Additive	Concentration of additive based on weight of slurry, percent	Brookfield apparent viscosity at 86° F, centipoises	Sedimentation volume, cc/g
Magnesium A			
A100	1.00	470	1.15
A101	1.00	210	1.26
A102	.3	3,300	1.68
A103	1.00	100,000-80,000	^b 1.9
A105	.16	4,120	1.92
A105	.79	600	1.43
A105	1.00	460	1.51
A105	1.75	690	1.27
A105	4.00	750	1.28
A106	1.00	3,180	1.89
A108	.50	2,690	1.93
A109	1.00	210	1.30
A110	1.00	200	1.06
A111	1.00	100,000-80,000	^b 1.9
A113	1.0	560	1.27
A116	1.00	2,550	1.91
A117	1.00	2,220	1.80
A118	.10	2,800	1.90
A118	.50	510	1.37
A118	1.00	230	1.10
A118	2.00	220	1.03
A118	4.00	170	1.00
A119	1.00	510	1.52
A120	1.00	2,850	1.90
A121	1.00	2,280	1.85
A123	1.00	100,000-80,000	^b 1.9
A124	1.00	350	1.07
A125	1.00	750	1.54
A127	.11	3,000	1.94
A127	.50	400	1.30
A127	1.01	870	1.46
A127	2.07	8,320	1.75
A132	.10	2,760	1.90
A132	.54	340	1.20
A132	1.00	200	1.02
A132	2.01	210	.99
A135	1.02	500	1.20
A136	.10	5,350	1.90
A136	.50	700	1.30
A136	1.00	720	1.30
A136	2.00	630	1.30
A137	1.00	220	1.03

^aSlurries made by standard procedure.^bEstimated. Slurry was too thick to be poured into graduated cylinders. Extremely little or no settling was observed in pint can.

TABLE VII. - Concluded. SEDIMENTATION VOLUMES AND BROOKFIELD APPARENT
VISCOSITIES OF SLURRIES MADE WITH MAGNESIUM A AND MAGNESIUM C^a

Additive	Concentration of additive based on weight of slurry, percent	Brookfield apparent viscosity at 86° F, centipoises	Sedimentation volume, cc/g
Magnesium A			
Al39	0.10	4,330	----
Al39	.2	1,400	----
Al39	.5	1,230	----
Al39	1.0	1,590	1.29
Al39	2.0	2,370	1.44
Al41	1.00	210	1.11
Al42	1.00	320	1.35
Al47	1.00	250	1.26
None	0	100,000-30,000	^b 1.9
Magnesium C			
Al04	1.00	15,000-10,000	^b 1.9
Al05	.10	4,480	1.90
Al05	.50	1,630	1.50
Al05	1.00	2,150	1.49
Al05	2.00	2,480	1.47
Al07	1.00	2,650	1.44
Al12	1.00	3,250	1.83
Al14	1.00	940	----
Al18	.10	3,500	1.90
Al18	.50	1,580	1.35
Al18	1.00	280	1.37
Al24	.10	4,080	1.93
Al24	.50	1,180	1.48
Al24	1.00	1,340	1.33
Al24	2.00	1,780	1.34
Al26	1.00	2,500	----
Al28	1.00	660	1.33
Al29	1.00	500	1.33
Al30	1.00	400	1.12
Al31	1.00	820	1.37
Al32	.20	2,130	1.58
Al32	.20	2,020	1.59
Al32	1.00	500	1.18
Al33	1.00	2,830	1.85
Al34	1.00	580	1.10
Al36	1.00	1,460	1.29
Al38	1.00	2,150	1.47
Al40	1.00	1,090	1.19
Al46	1.00	430	1.35
None	0	100,000-20,000	^b 1.9

^aSlurries made by standard procedure.

^bEstimated. Slurry was too thick to be poured into graduated cylinders. Extremely little or no settling was observed in pint can.

TABLE VIII. - COMPARISON OF ADDITIVES ON BASIS OF BROOKFIELD
APPARENT VISCOSITY OF SLURRY

Range	Additive	Concentration of additive based on weight of slurry, percent	Quantity of additive per 400 g of slurry, millimoles	Brookfield apparent viscosity at 86° F, centipoises		
				Magnesium A	Magnesium B	Magnesium C
1	A132	1.00 ↓	2.7	200	400	500
	A110		11.2	200		
	A109		12	210		
	A101		----	210		
	^a A141		----	210		
	A137		9.3	220		
	A118		5.1	230		
	^a A147		---	250		280
2	A142	1.00	4.2	320	720 960	1340 2150 1460
	A124	1.00	14.5	350		
	A127	^b 0.50	7.1	400		
	A105	1.00	16.5	460		
	^a A100	1.00 ↓	10.3	470		
	A135		----	500		
	A119		6.7	510		
	A113		---	560		
	A136		9.0	720		
	A125		30.8	750		
	A139	^b 0.50	7.0	1230		
	A117	1.00	---	2220		
	A121	1.00	24.2	2280		
	A116	1.00	----	2550		
	A108	^b 0.50	13.9	2690		
	A120	1.00	19.1	2850		
	A106	1.00	14	3180		
	^a A102	^c 0.3	3.6	3300		
3	None	0	0	100,000-30,000	100,000-25,000	100,000-20,000
	A111	1.00 ↓	24.2	100,000-80,000		
	A123		32.5	100,000-80,000		
	A103		----	100,000-80,000		

^aData for this additive not shown in table IX.

^bLess than 1.00 percent used because fig. 6(b) indicates minimum viscosity obtained with approximately 0.5 percent of carboxylic acid.

^cLess than 1.00 percent used to avoid gelation of hydrocarbon.

TABLE VIII. - Concluded. COMPARISON OF ADDITIVES ON BASIS OF
BROOKFIELD APPARENT VISCOSITY OF SLURRY

Range	Additive	Concentration of additive based on weight of slurry, percent	Quantity of additive per 400 g of slurry, millimoles	Brookfield apparent viscosity at 86° F, centipoises
Magnesium B				
1	^a A148	1.00	----	390
	A132	↓	2.7	400
	^a A149		----	490
2	A115	1.00	----	510
	^a A144	↓	----	590
	A142		4.2	720
	A105		16.5	890
	^a A145	↓	----	900
3	A122	1.00	13 (approx.)	70,000-22,000
	A143	↓	22.5	65,000-17,000
	None	0	0	100,000-25,000
Magnesium C				
1	A118	1.00	5.1	280
	A130	↓	7.4	400
	^a A146		----	430
	A132		2.7	500
	A129		11	500
	A134		8.5	580
	A128	↓	15	660
2	A131	1.00	11	820
	A114	↓	----	^b 940
	A140		6 (approx.)	1090
	A124		14.5	1340
	A136		9.0	1460
	A138		4.5	2150
	A105		16.5	2150
	A126	↓	----	2500
	A107		7.0	2650
	A133		8.2	2830
	A112	↓	----	3250
3	A104	1.00	30.8	15,000-10,000
	None	0	0	100,000-20,000

^aData for this additive not shown in table IX.

^bViscosity of 590 centipoises was obtained once for this slurry, but was disregarded because it was inconsistent with previous and subsequent measurements.

TABLE IX. - GROUPING OF ADDITIVES BY VISCOSITY RANGE
AND TYPES OF POLAR GROUPS

Polar groups in molecules of additive	Range 1			Range 2			Range 3		
	Magnesium			Magnesium			Magnesium		
	A	B	C	A	B	C	A	B	C
Ester or metal salt				A113 A116 A117 A135 A136 A142	A115 A142	A114 A136 A138 A140			
Hydroxyl or carboxyl				A105 A108 A119 A120 A121 A125 A127 A139	A105	A105 A126	A103		
Hydroxyl plus one or more of following: Ester Metal salt of carboxylic acid Polyoxyethylene	A101 A109 A110 A118 A132 A137	A132	A118 A128 A129 A130 A132 A134			A112 A131 A133			
Primary amine				A106 A124		A124		A122	
Other						A107	A111 A123	A143	A104

TABLE X. - BROOKFIELD APPARENT VISCOSITIES OF SLURRIES
CONTAINING TWO SURFACE-ACTIVE ADDITIVES

Additives, percent by weight		Brookfield apparent viscosity, centipoises	Additives, percent by weight		Brookfield apparent viscosity, centipoises
Magnesium B			Magnesium C		
Al24	Al32		Al32	Al36	
0	1.00	400	0.05	0.05	4360
.10	.90	360	.10	.10	3070
.20	.80	340	.25	.25	730
.50	.50	360	.50	.50	1070
.80	.20	740			
.90	.10	790	1.00	0	500
1.00	0	(a)	0	1.00	1460

^aSlurry with 1.00 percent Al24 not prepared with magnesium B. Viscosities of slurries made with 1.00 percent Al24 and magnesium A or magnesium C are listed in table VIII.



Figure 1.- Electron micrograph of 1.5-micron magnesium powder.

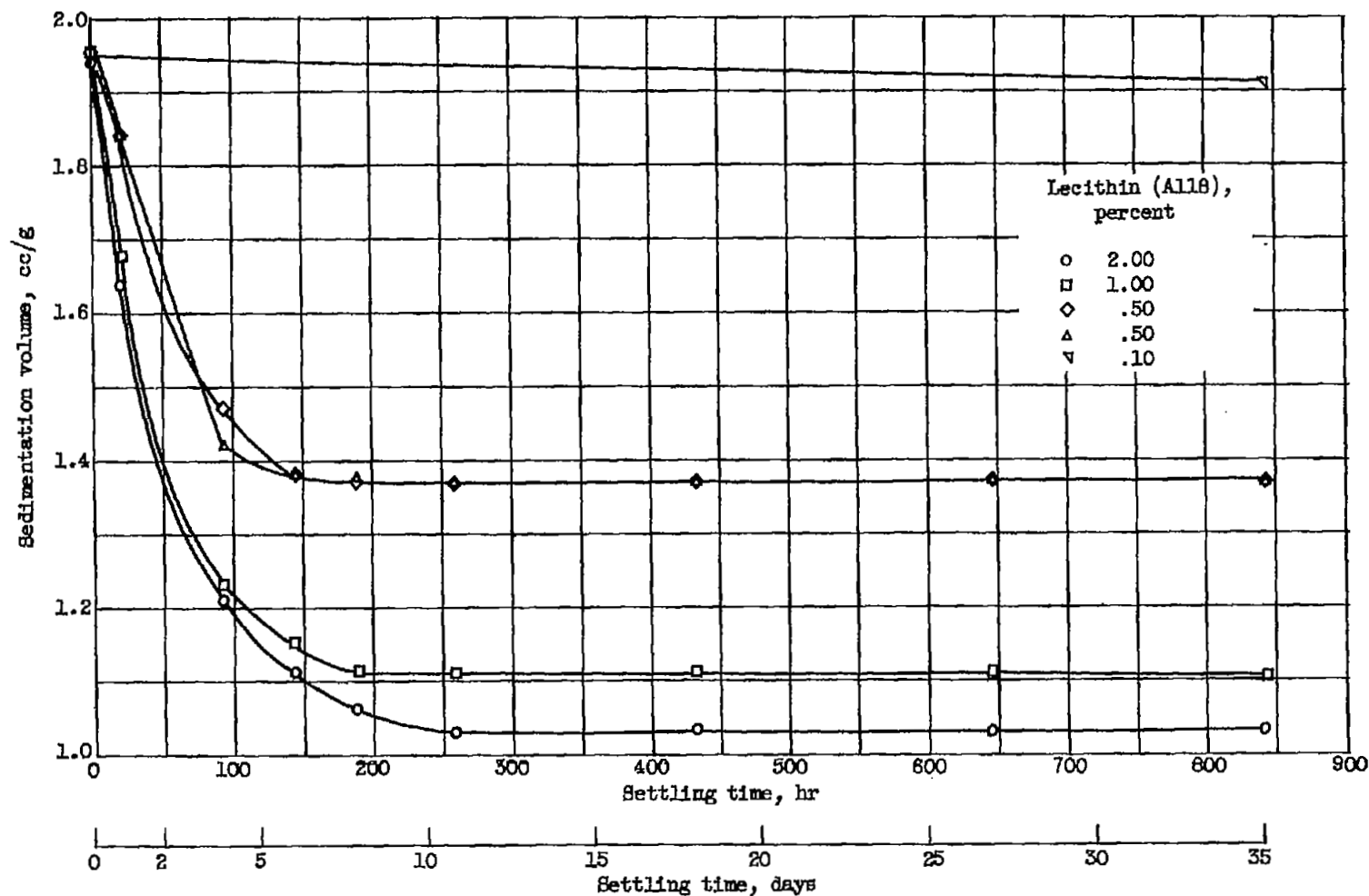


Figure 2. - Typical family of settling curves for magnesium slurries over a range of concentrations of lecithin. Slurries contain 50 percent magnesium A; standard procedure.

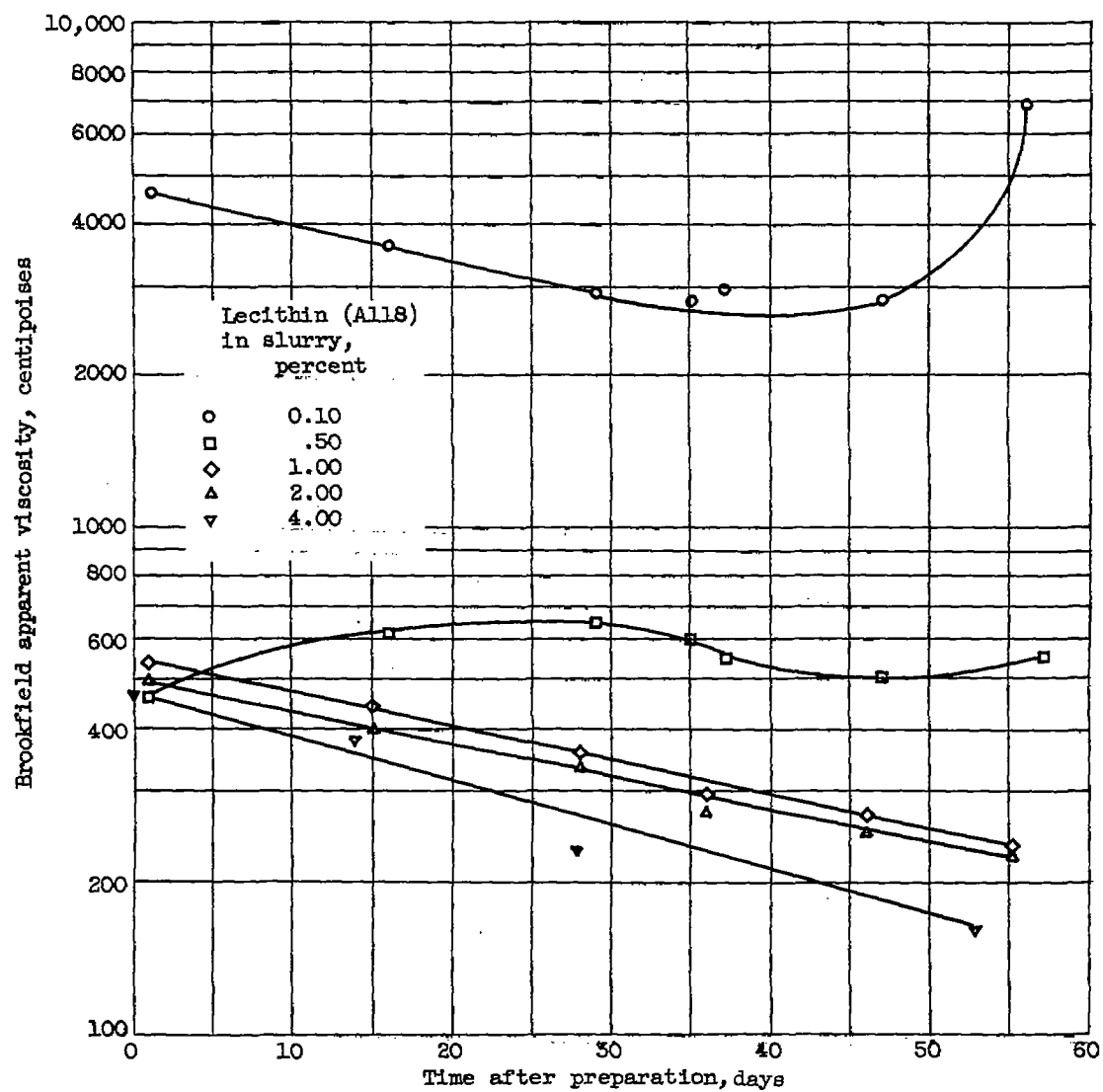


Figure 3. - Effect of aging on Brookfield apparent viscosity of magnesium slurries containing various concentrations of lecithin (All8). Slurries contain 50 percent magnesium A; standard procedure.

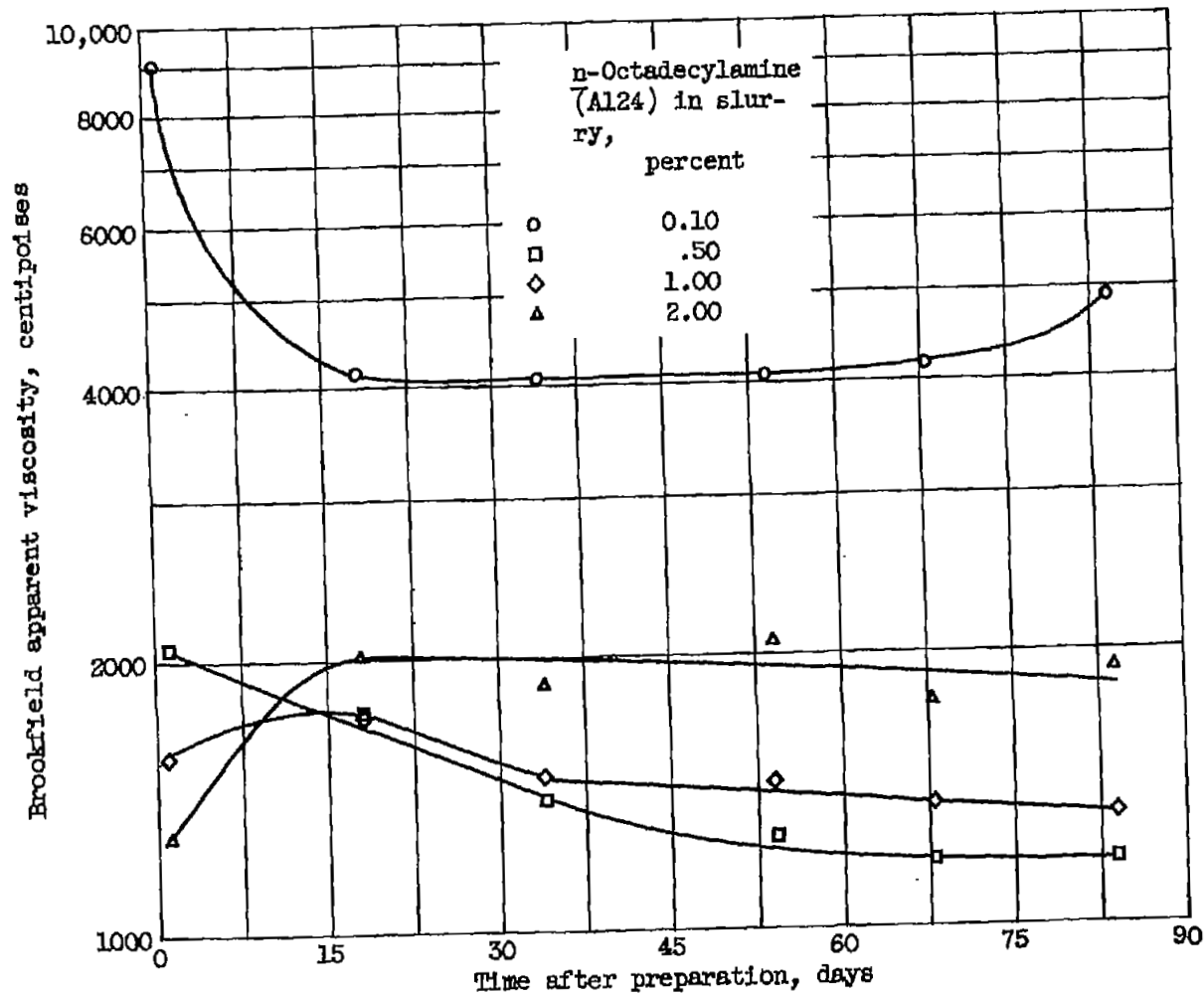


Figure 4. - Effect of aging on Brookfield apparent viscosity of magnesium slurries containing various concentrations of n-octadecylamine (Al24). Slurries contain 50 percent magnesium C; standard procedure

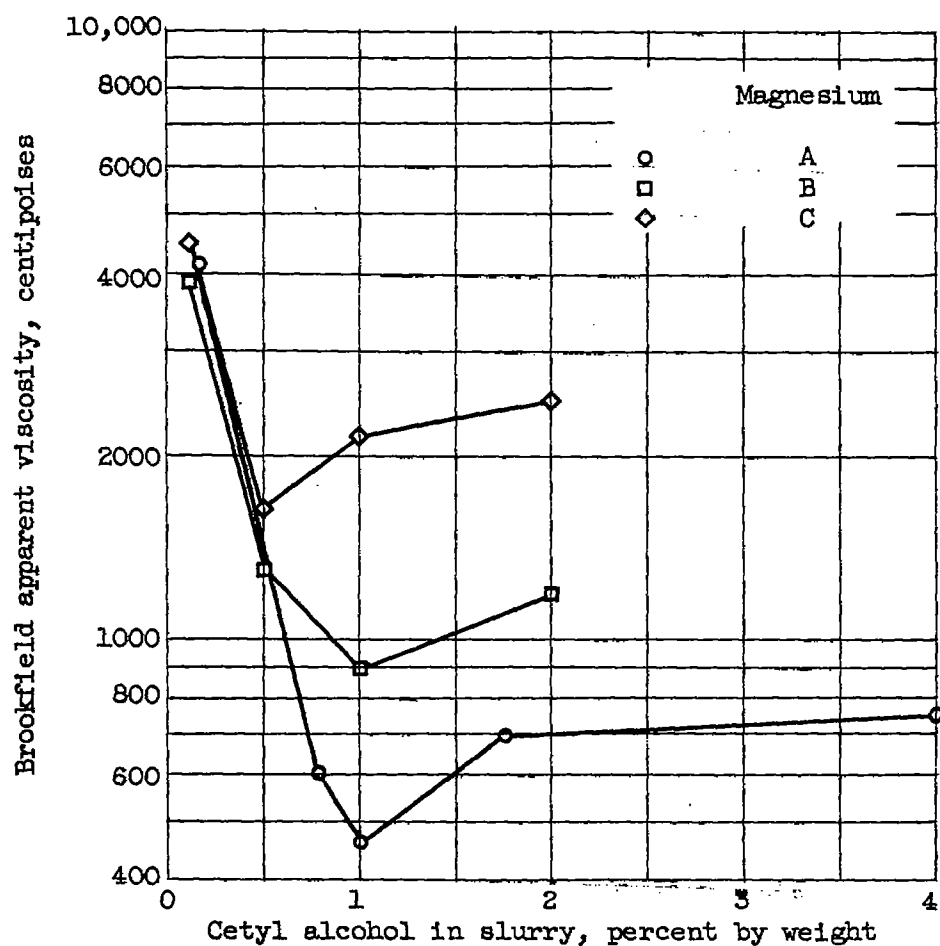
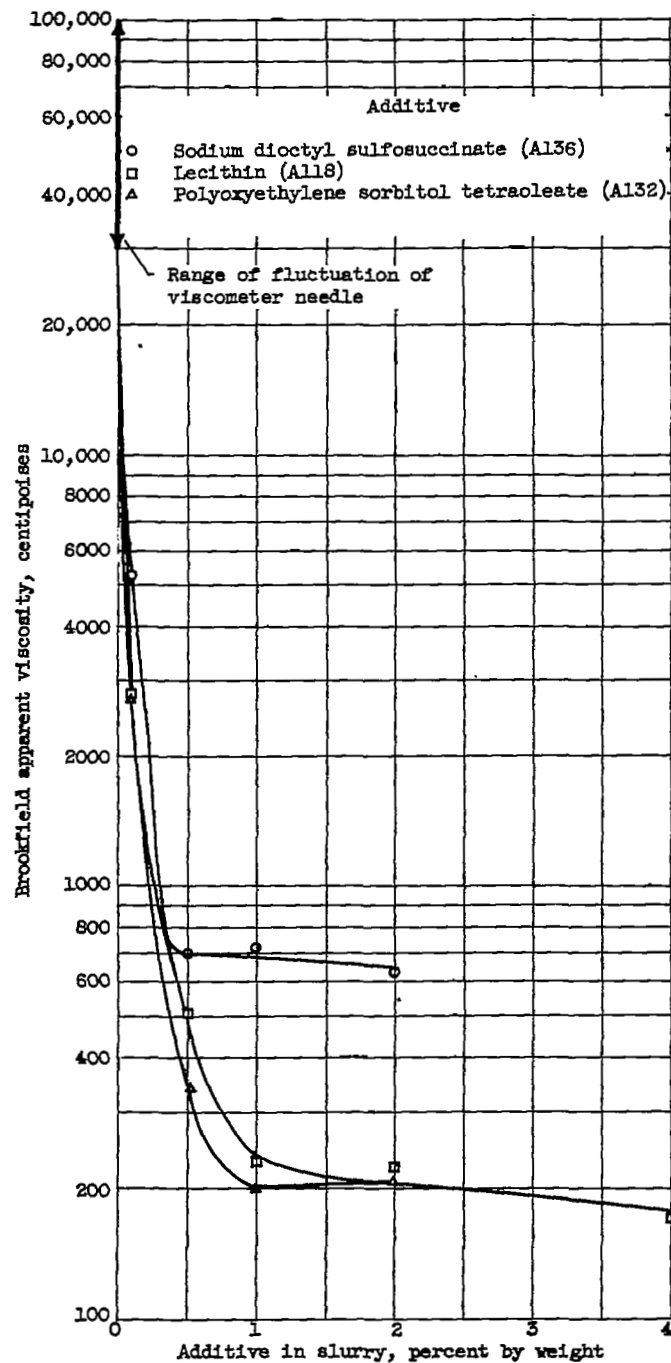
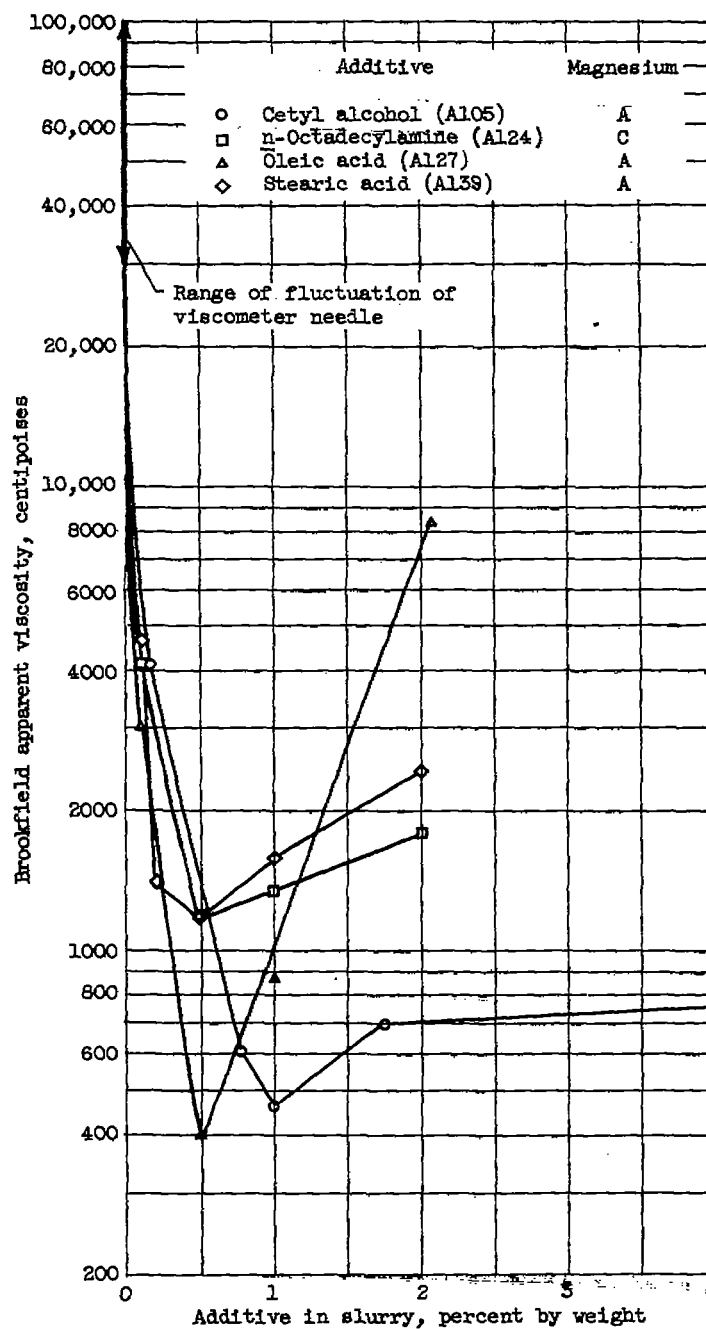


Figure 5. - Comparison of Brookfield apparent viscosities of three slurries made with magnesium powder from different drums and containing various percentages of cetyl alcohol (Al05). Slurries contain 50 percent magnesium; standard procedure.



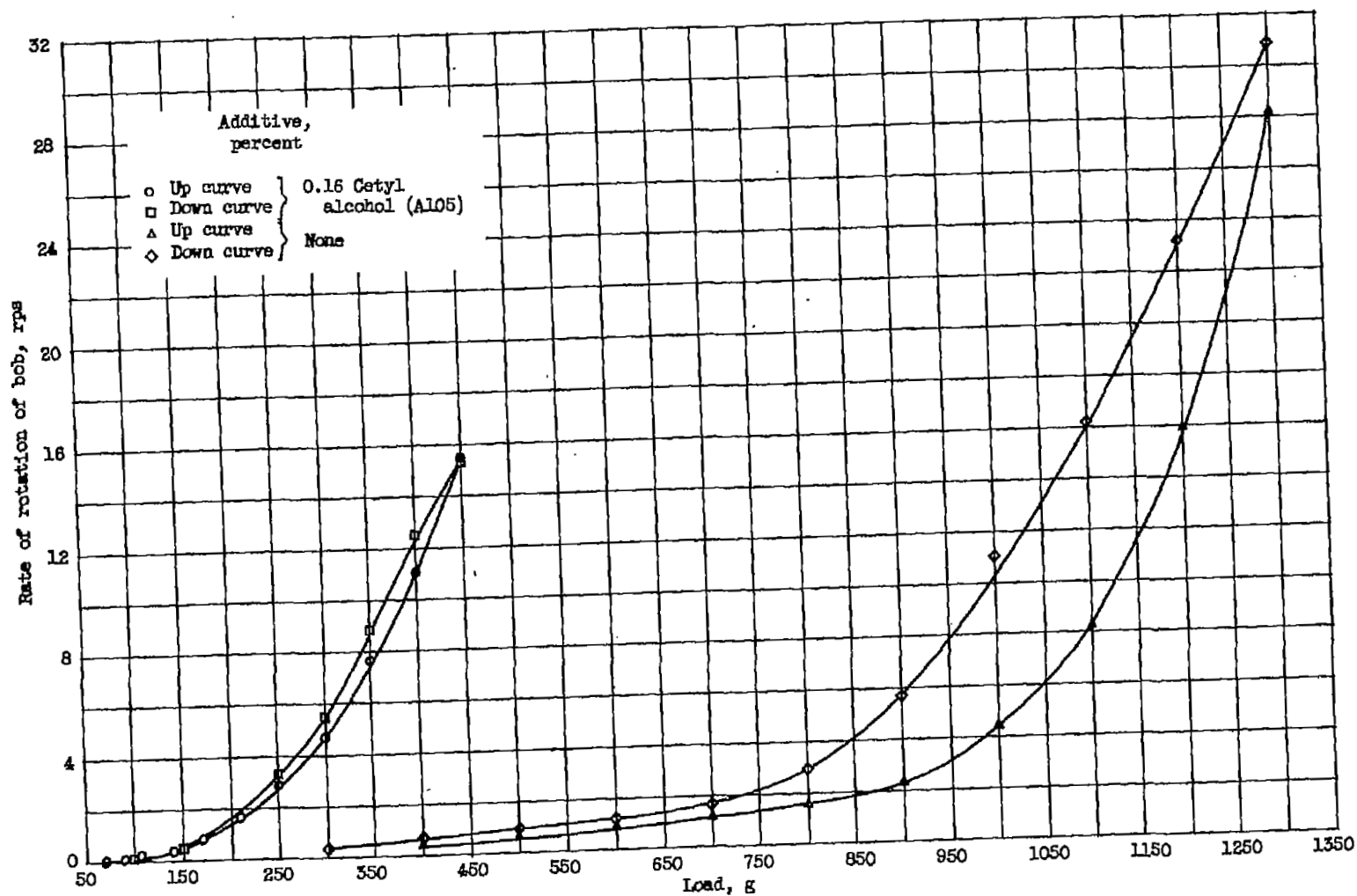
(a) Additive molecules are bulky and have multiple polar groups. Slurries contain 50 percent magnesium A; standard procedure.

Figure 8. - Effect of concentration of surface-active additives on Brookfield apparent viscosity of magnesium slurries.



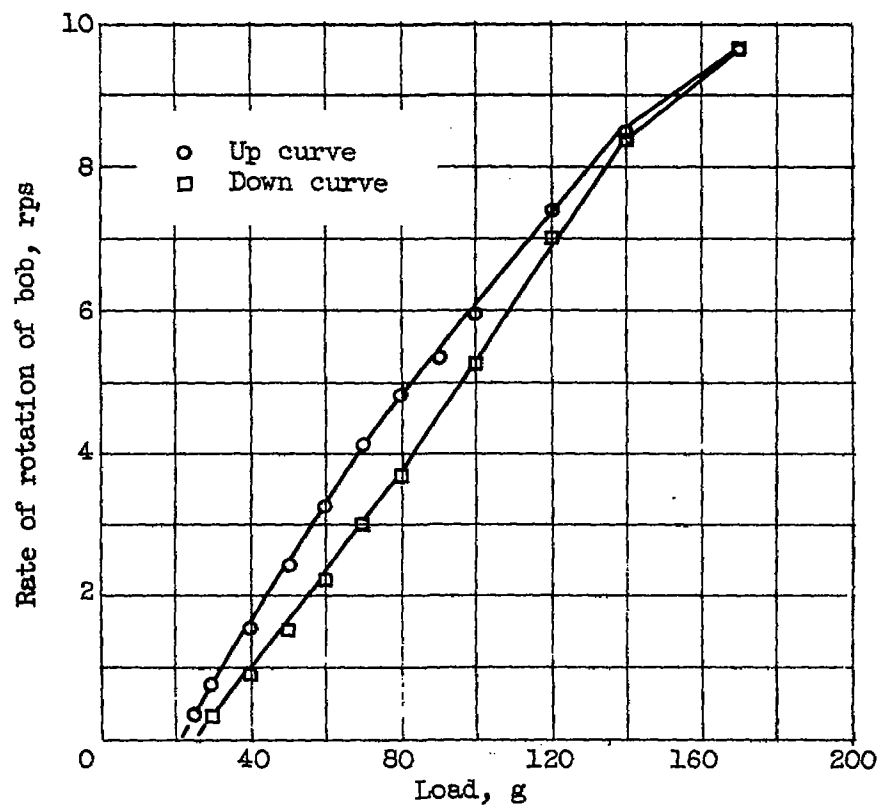
(b) Additive molecules are slender and each has only one polar group. Slurries contain 50 percent magnesium A or C; standard procedure.

Figure 6. - Concluded. Effect of concentration of surface-active additives on Brookfield apparent viscosity of magnesium slurries.



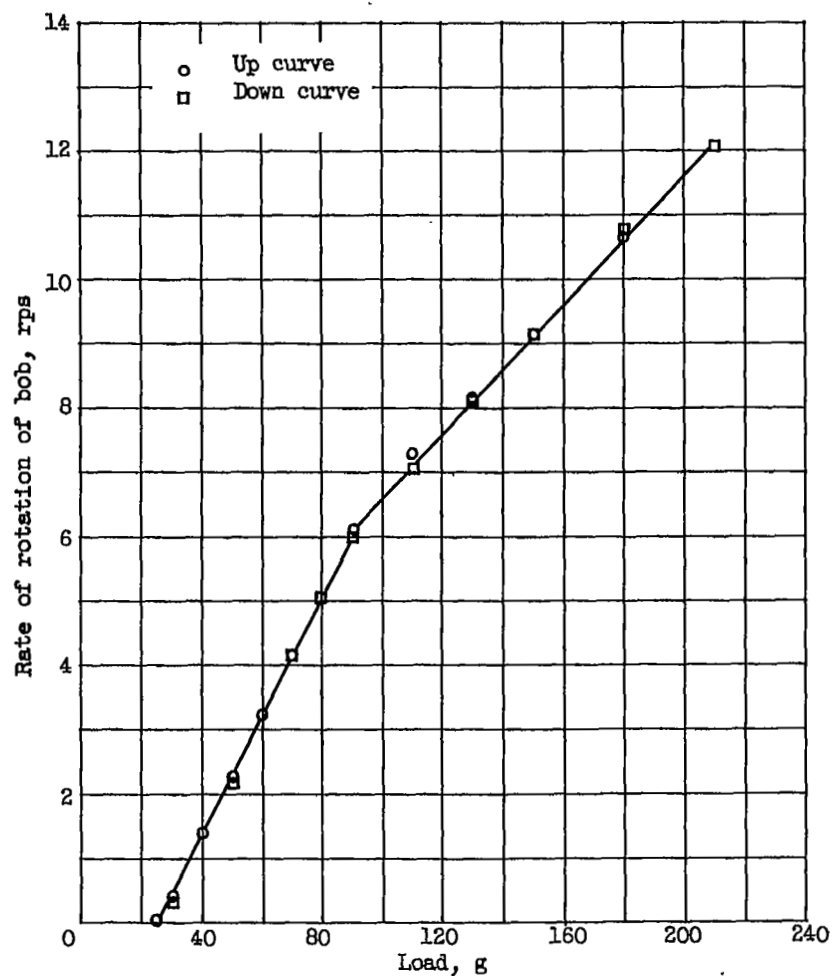
(a) Slurries with no additive and with 0.16 percent cetyl alcohol (A105). Run with cup and bob 2.

Figure 7. - Stormer flow curves for magnesium slurries. Slurries contain 50 percent magnesium A; standard procedure.

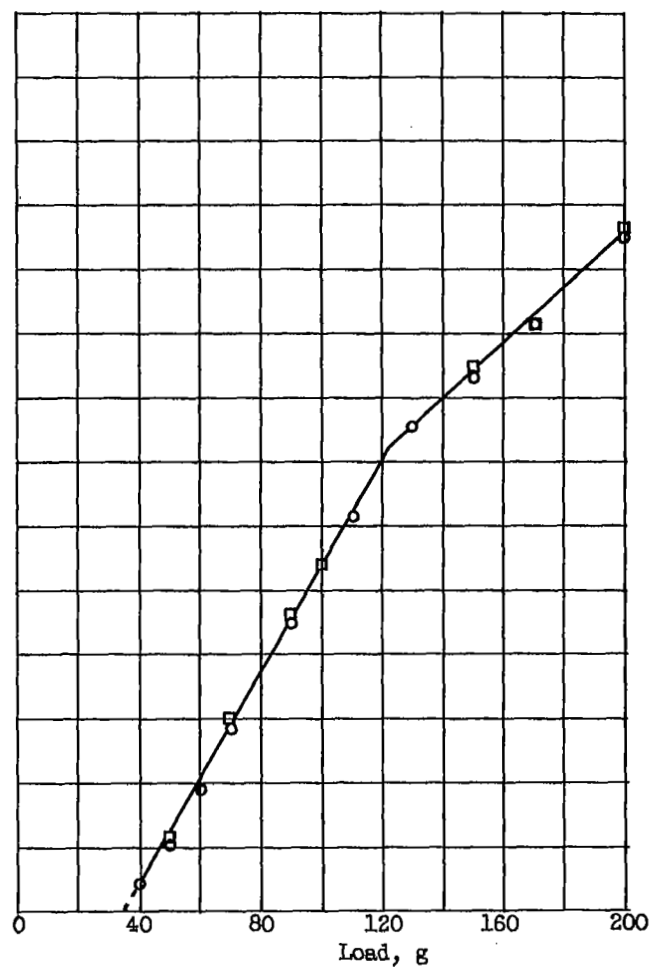


(b) Slurry with 0.79 percent cetyl alcohol. Run with cup and bob 2.

Figure 7. - Continued. Stormer flow curves for magnesium slurries. Slurries contain 50 percent magnesium A; standard procedure.



(c) Slurry with 1.75 percent cetyl alcohol. Run with cup and bob 1.



(d) Slurry with 4.00 percent cetyl alcohol. Run with cup and bob 1.

Figure 7. - Concluded. Stormer flow curves for magnesium slurries. Slurries contain 50 percent magnesium A; standard procedure.

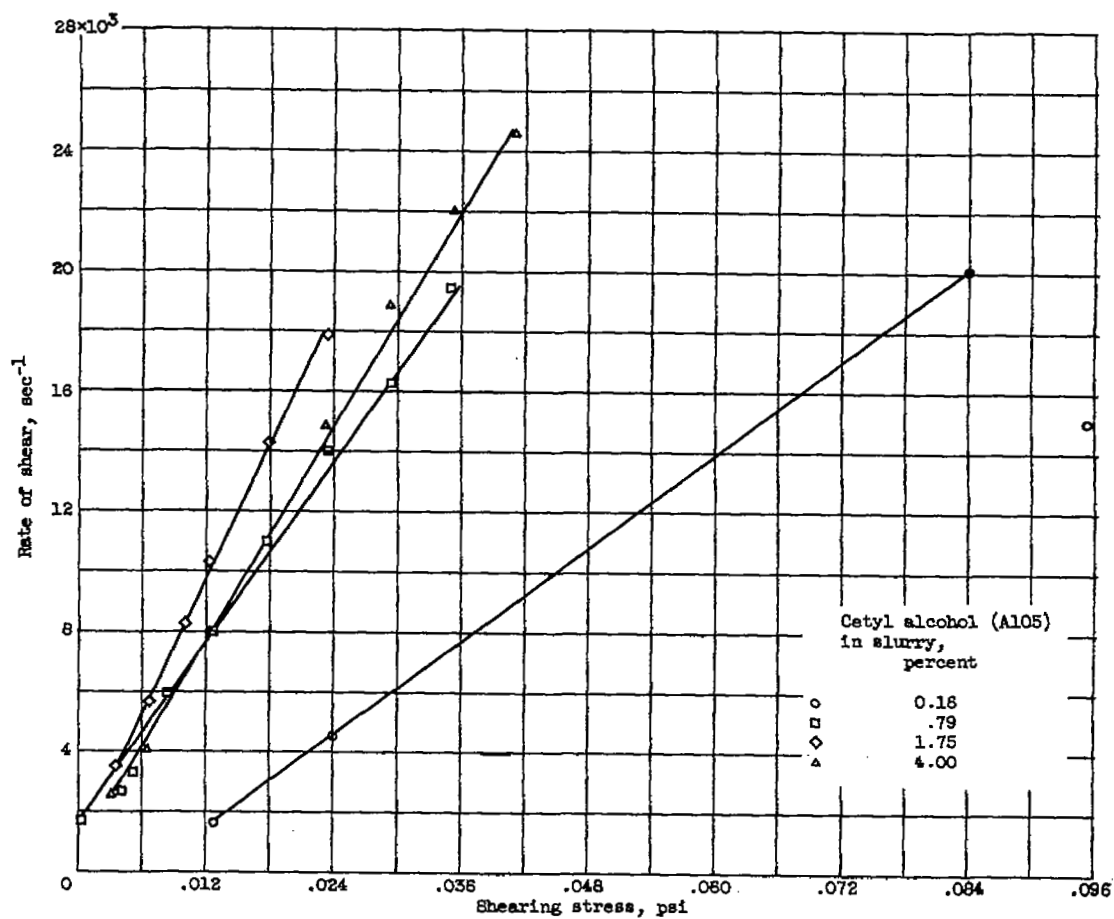


Figure 8. - Severs flow curves for magnesium slurries containing various concentrations of cetyl alcohol (Al05). Slurries contain 50 percent magnesium A; standard procedure.

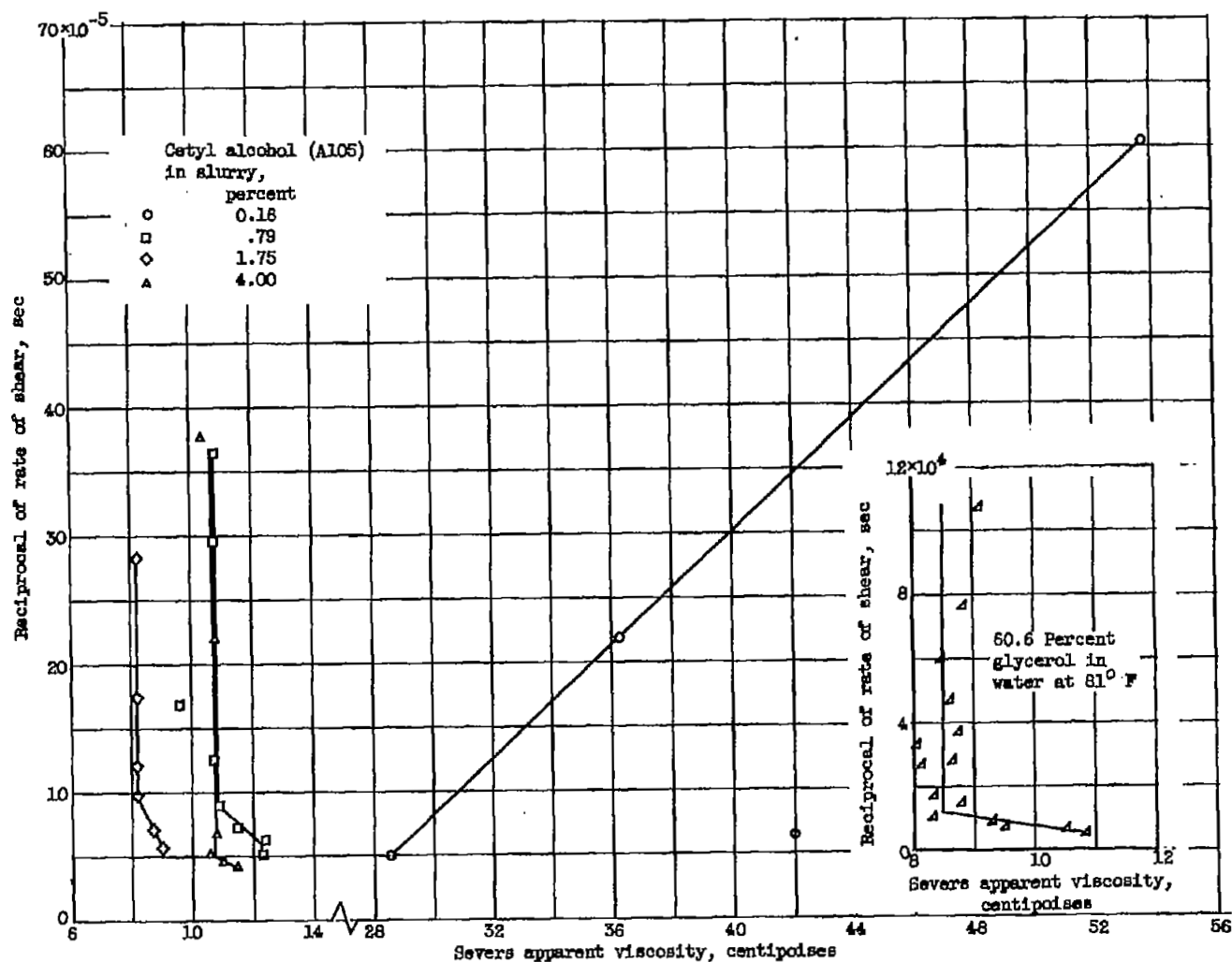


Figure 9. - Severs reciprocal of rate of shear against apparent viscosity for magnesium slurries containing various concentrations of cetyl alcohol (A105). Slurries contain 50 percent magnesium A; standard procedure. Inset shows similar type of plot for 60.6 percent glycerol in water.

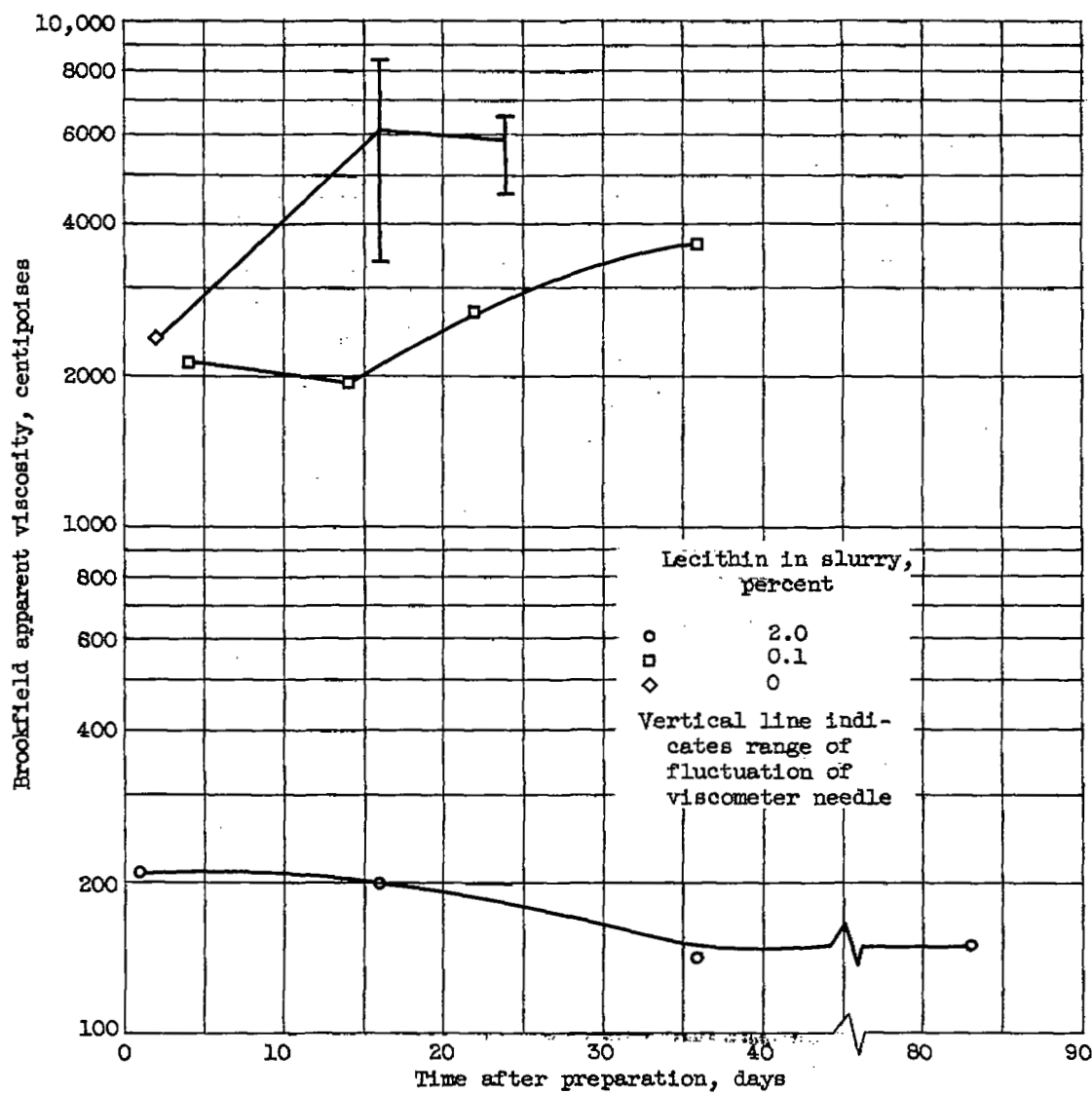


Figure 10. - Effect of aging on Brookfield apparent viscosity of magnesium slurries prepared by heating-flushing procedure and containing various concentrations of lecithin (All8). Slurries contain 50 percent magnesium A.

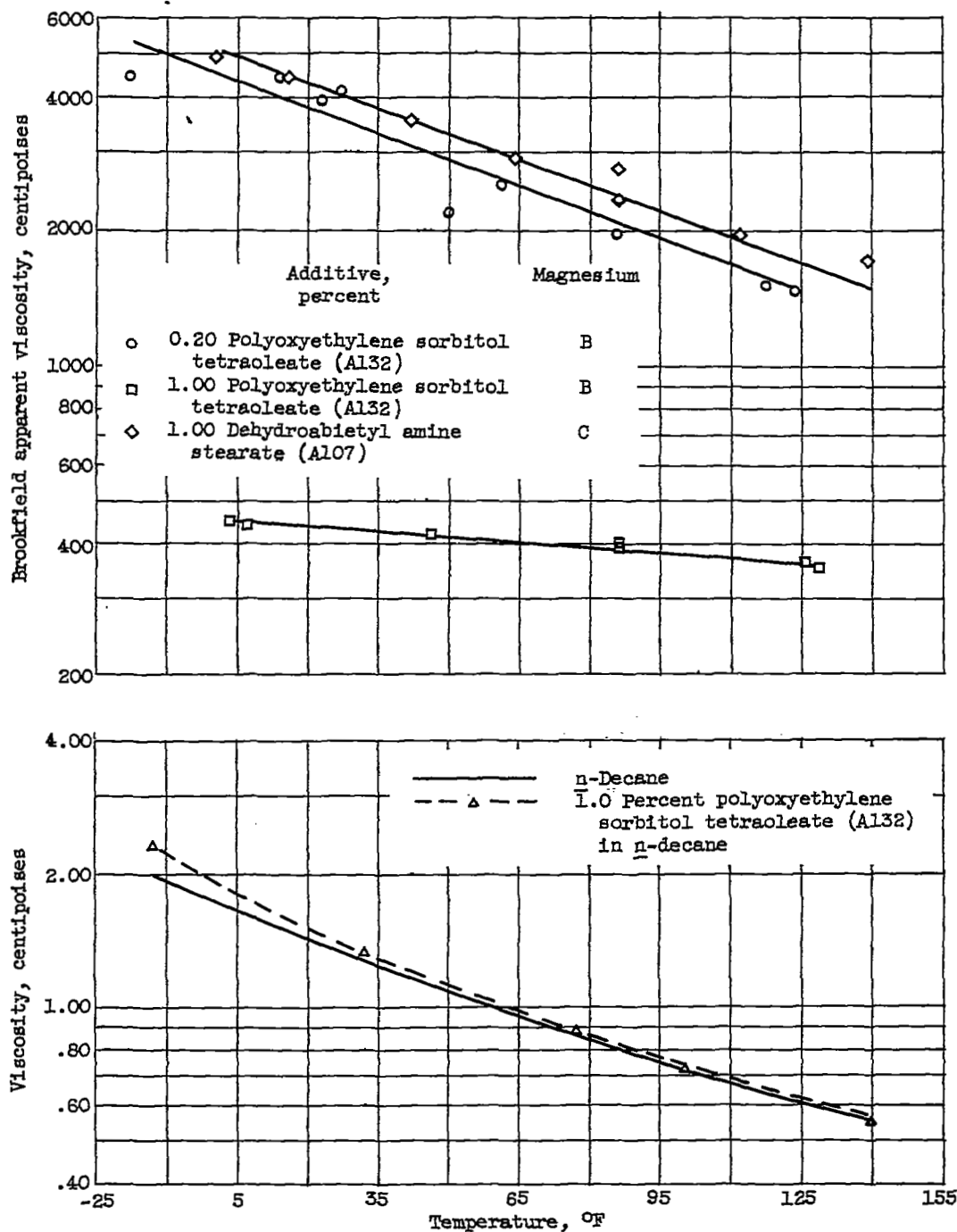


Figure 11. - Effect of temperature on Brookfield apparent viscosity of magnesium slurries containing different additives. Effect of temperature on viscosity of pure n-decane and n-decane containing 1.0 percent A132 is shown for comparison. Data for plot for n-decane were taken from reference 36.

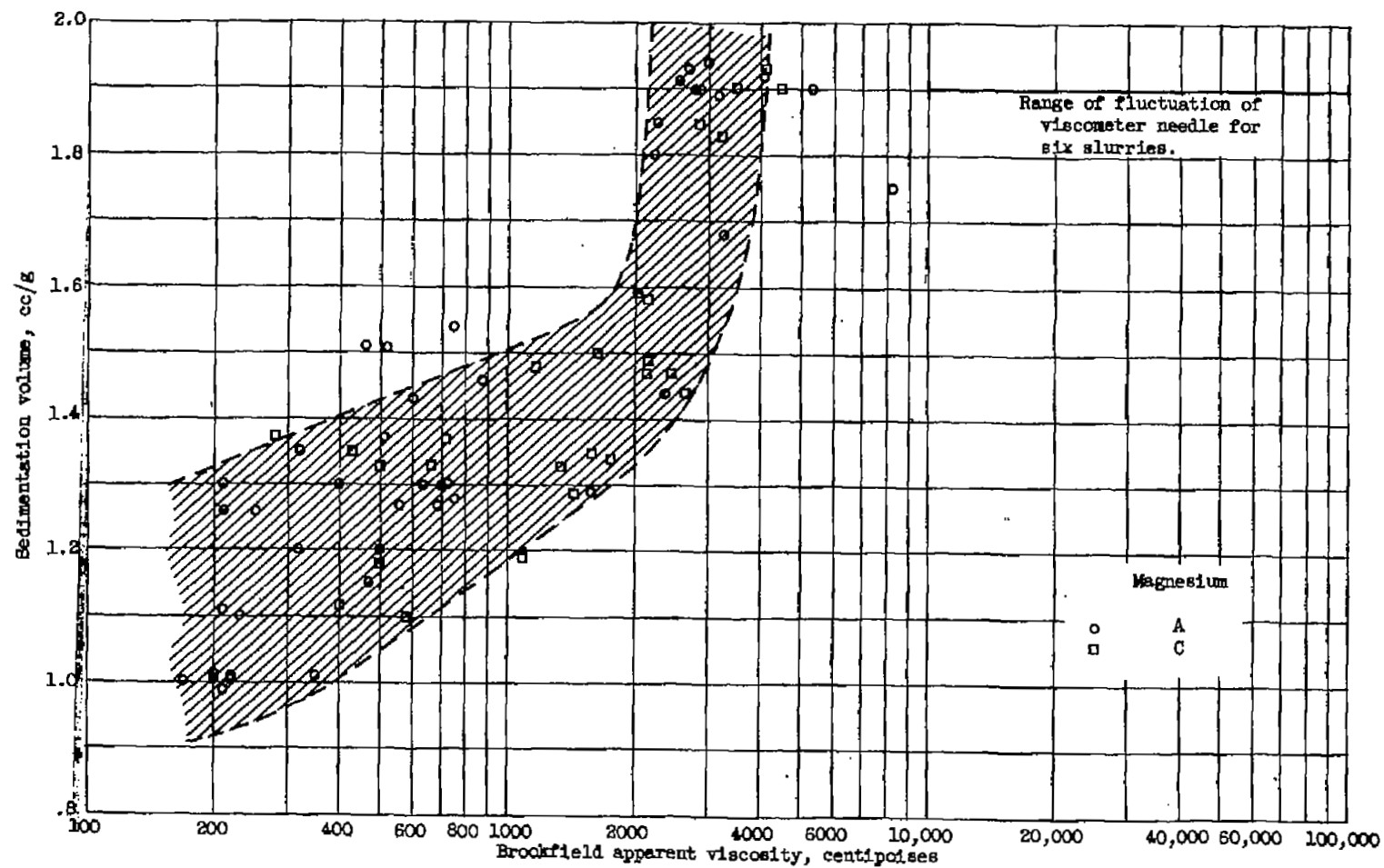


Figure 12. - Relation of sedimentation volume to Brookfield apparent viscosity. Slurries contain 50 percent magnesium; standard procedure.